Tappi

JOURNAL OF THE TECHNICAL ASSOCIATION OF THE PULP AND PAPER INDUSTRY

JANUARY 1960

VOLUME 43 • NUMBER 1

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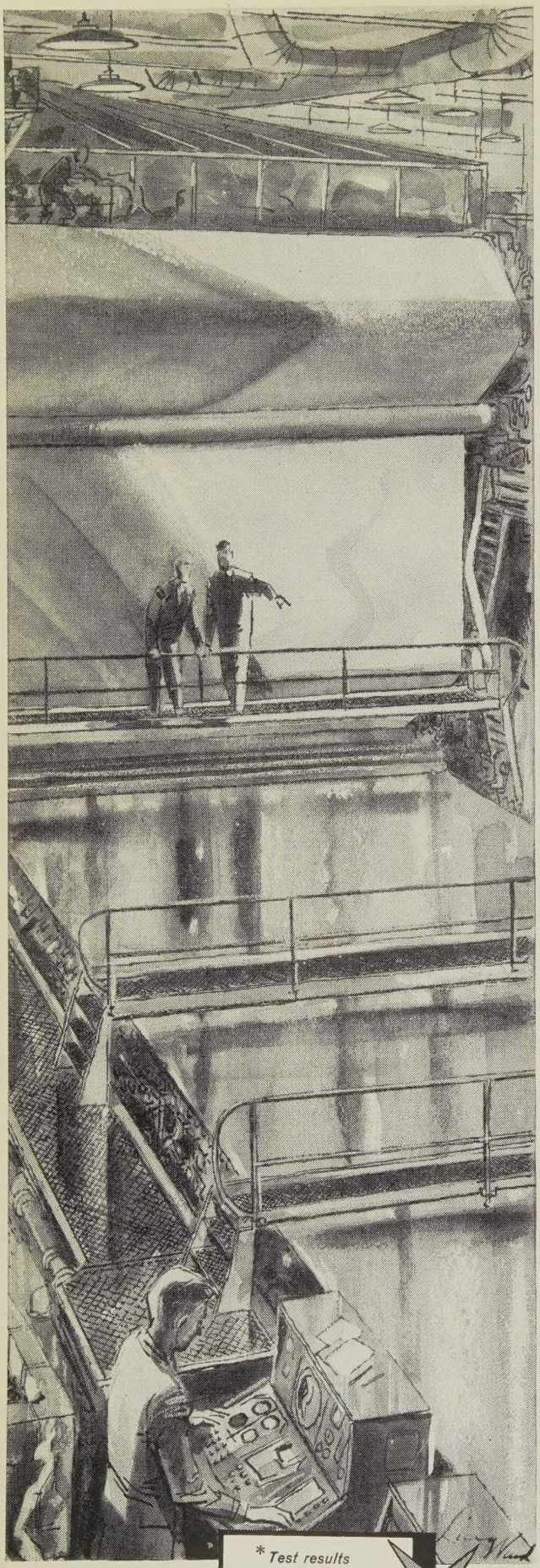

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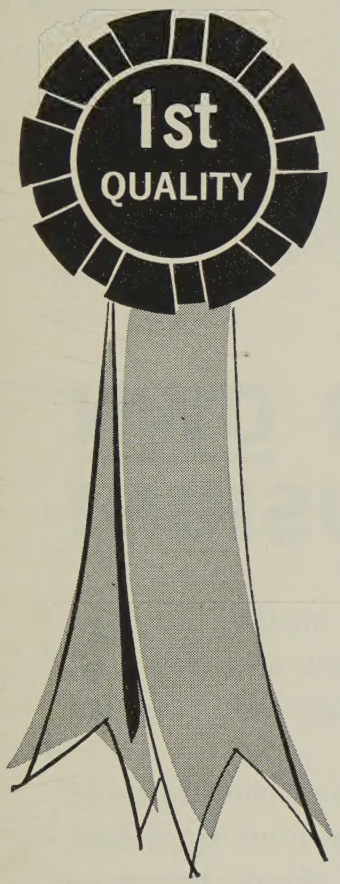

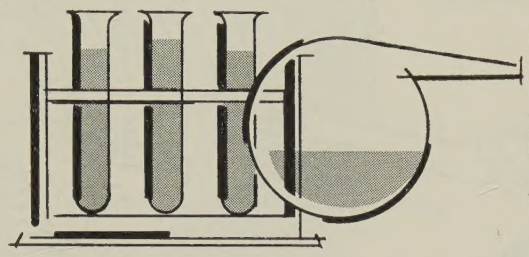

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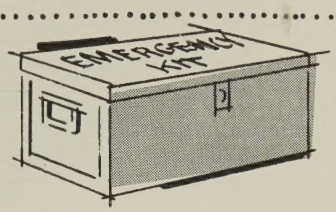

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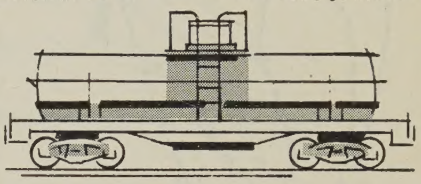

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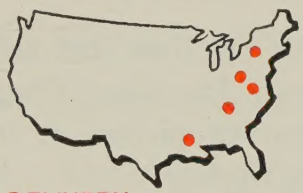

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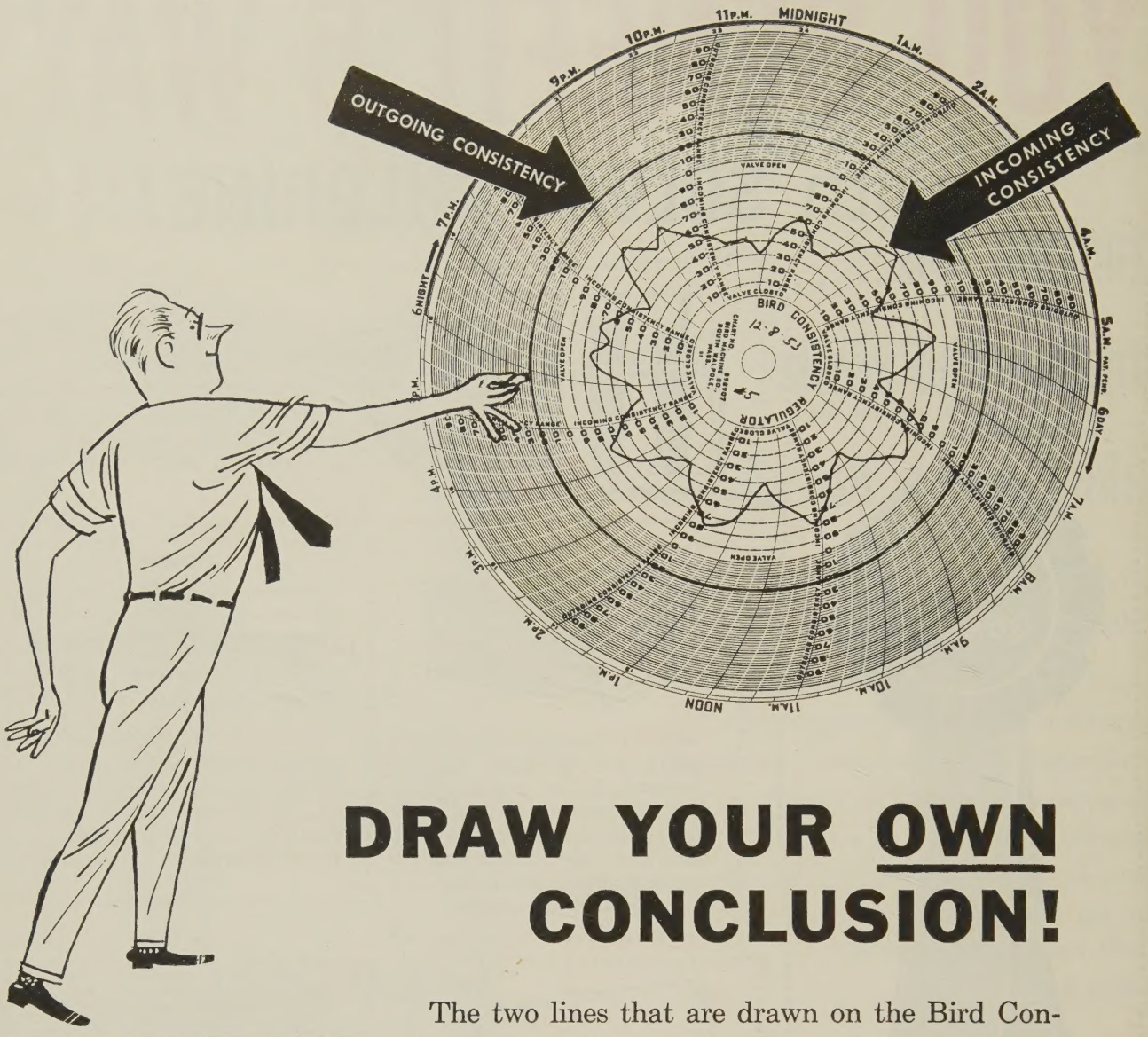

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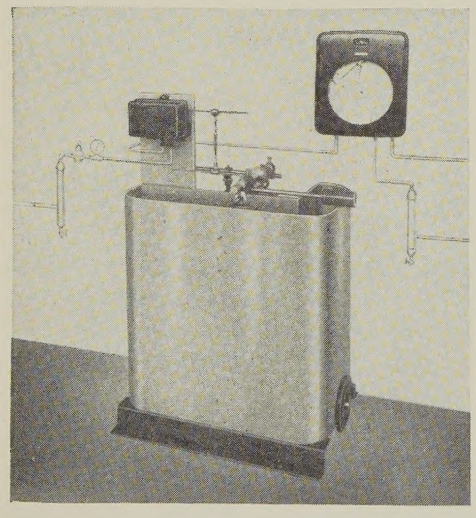

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Feb. 22–25, 1960.

Eleventh Coating Conference
Edgewater Beach Hotel, Chicago, Ill.
May 23–25, 1960.

Pulp Bleaching Conference
Edgewater Beach Hotel, Chicago, Ill.
June 14–16, 1960.

Fourteenth Alkaline Pulping Conference
Multnomah Hotel, Portland, Ore.
Aug. 22–24, 1960.

Forest Biology Conference
Seattle, Wash.
Aug. 24–27, 1960.

Eleventh Testing Conference
Pantlind Hotel, Grand Rapids, Mich.
Sept. 27–29, 1960.

Tenth Corrugated Containers Conference
Royal York Hotel, Toronto, Canada
Oct. 10–13, 1960.

Fifteenth Plastics-Paper Conference
Hotel Syracuse, Syracuse, N. Y.
Oct. 17–19, 1960.

Fifteenth Engineering Conference
Robert Meyer Hotel, Jacksonville, Fla.
Oct. 24–28, 1960.

Please refer to Local Section Activities for the locations and dates of Section meetings

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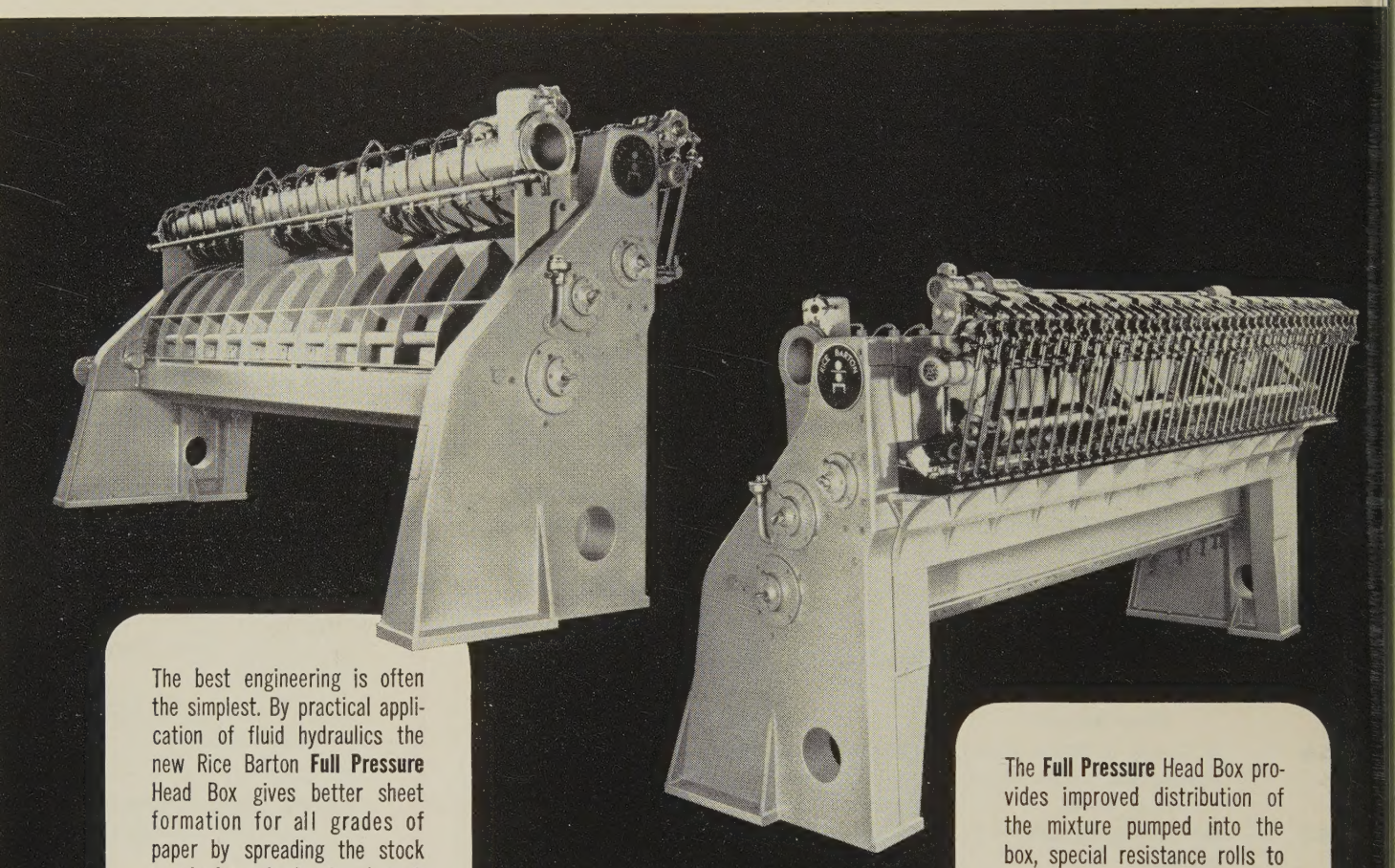

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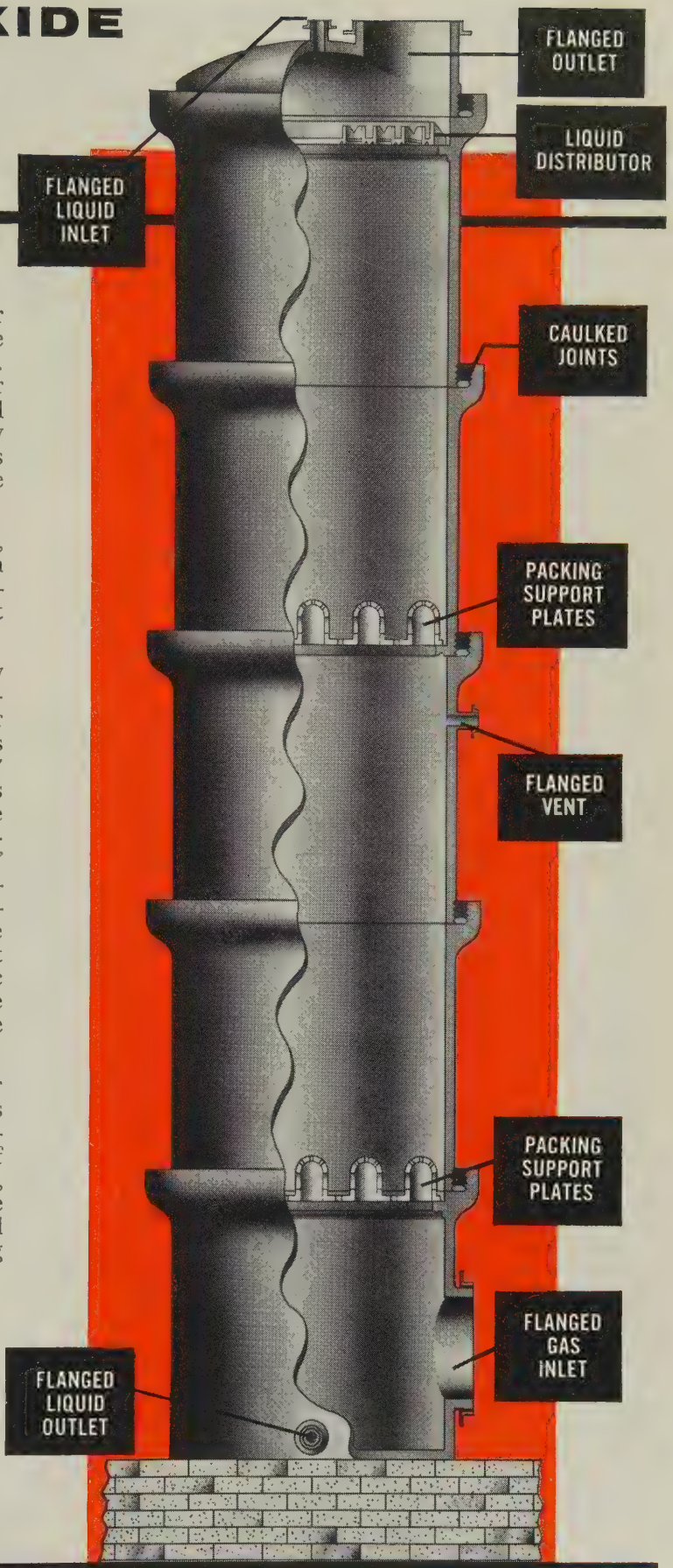

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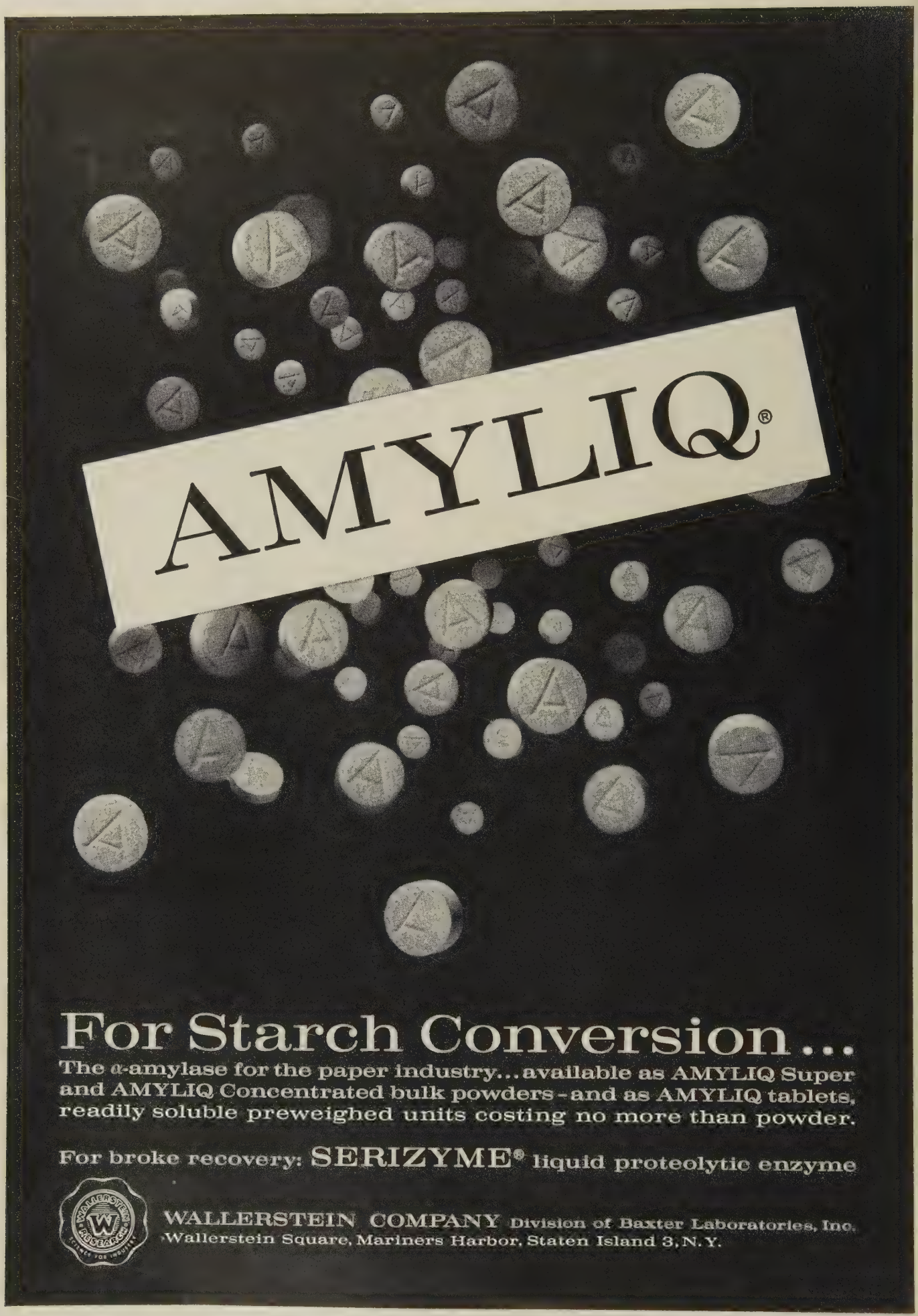

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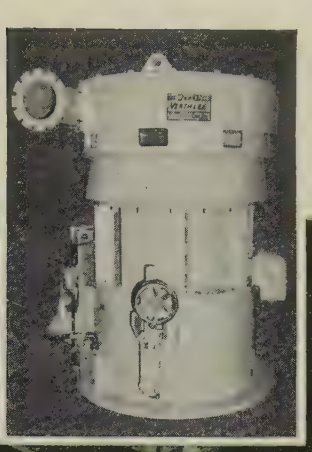

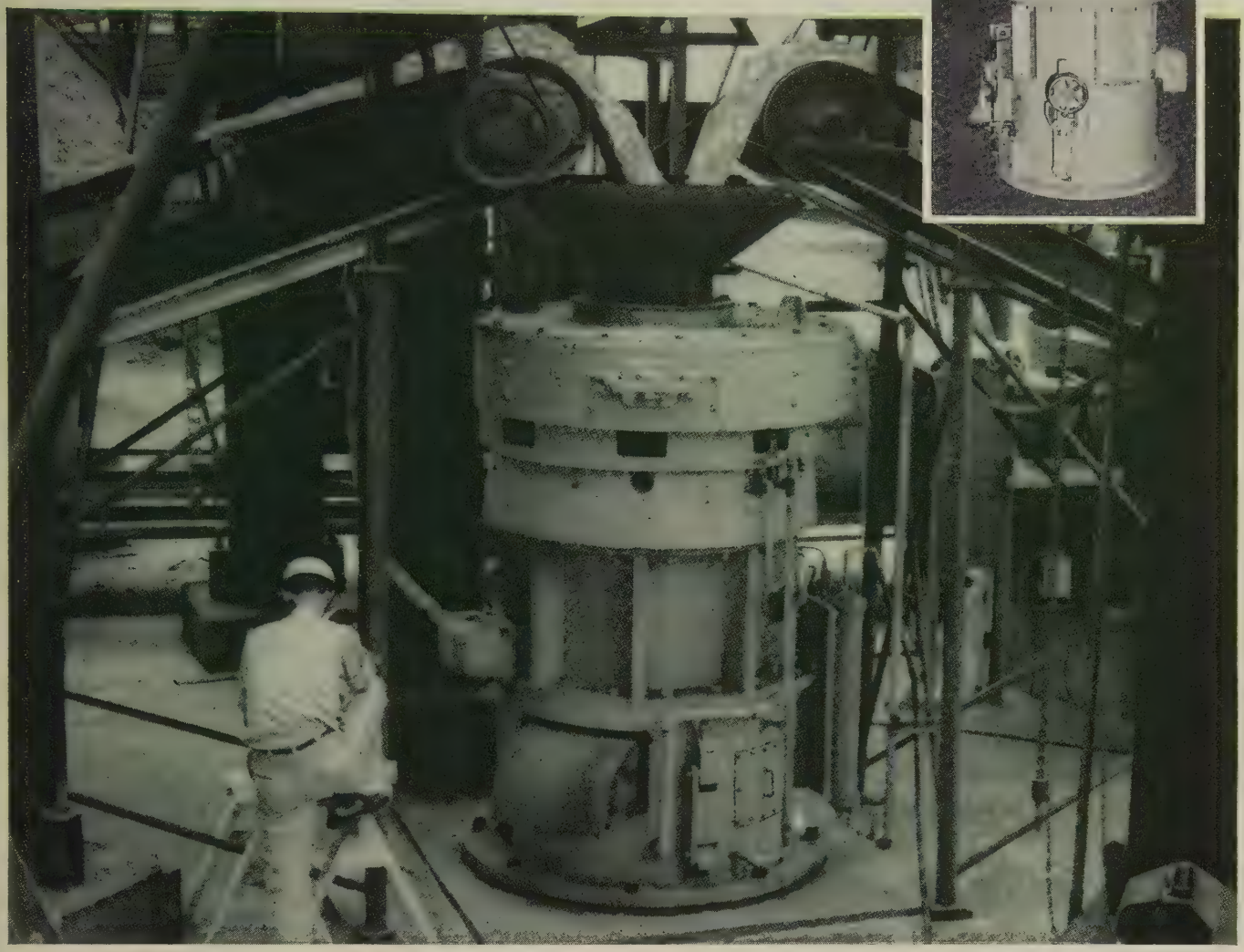

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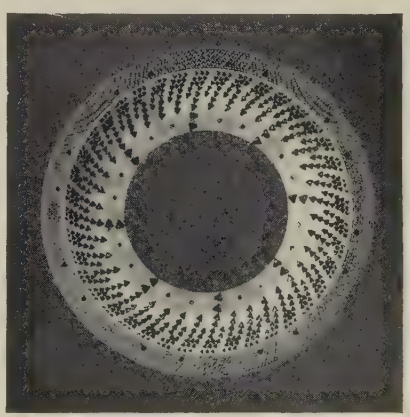

Teeth in vertically mounted rotating disc inter-mesh with teeth in opposing stationary plate mounted in Vertiflex cover. Clearance is easily adjustable and disc life is high since there is never any metal-to-metal contact. Teeth shape and arrangements are varied to fit the application.

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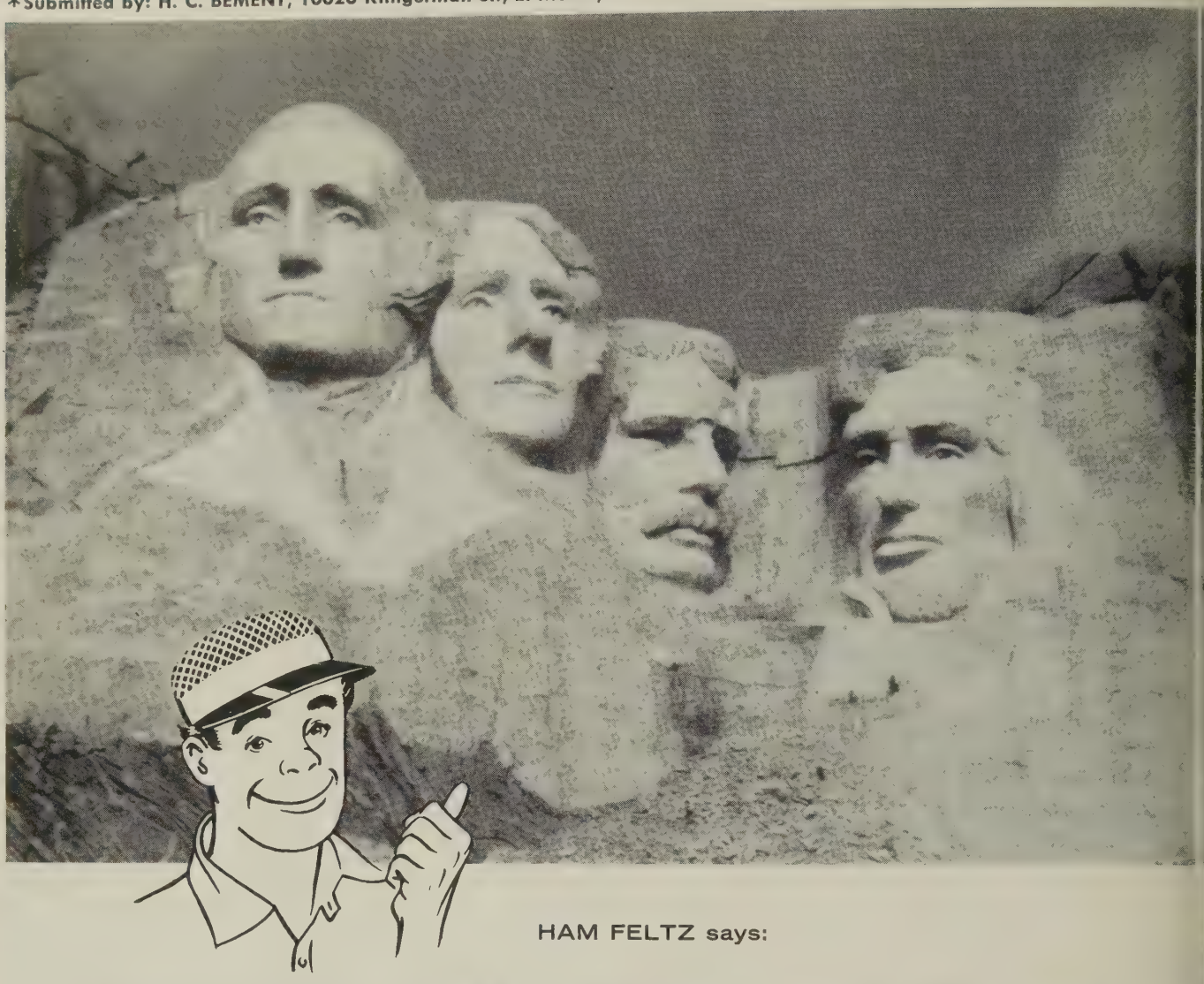

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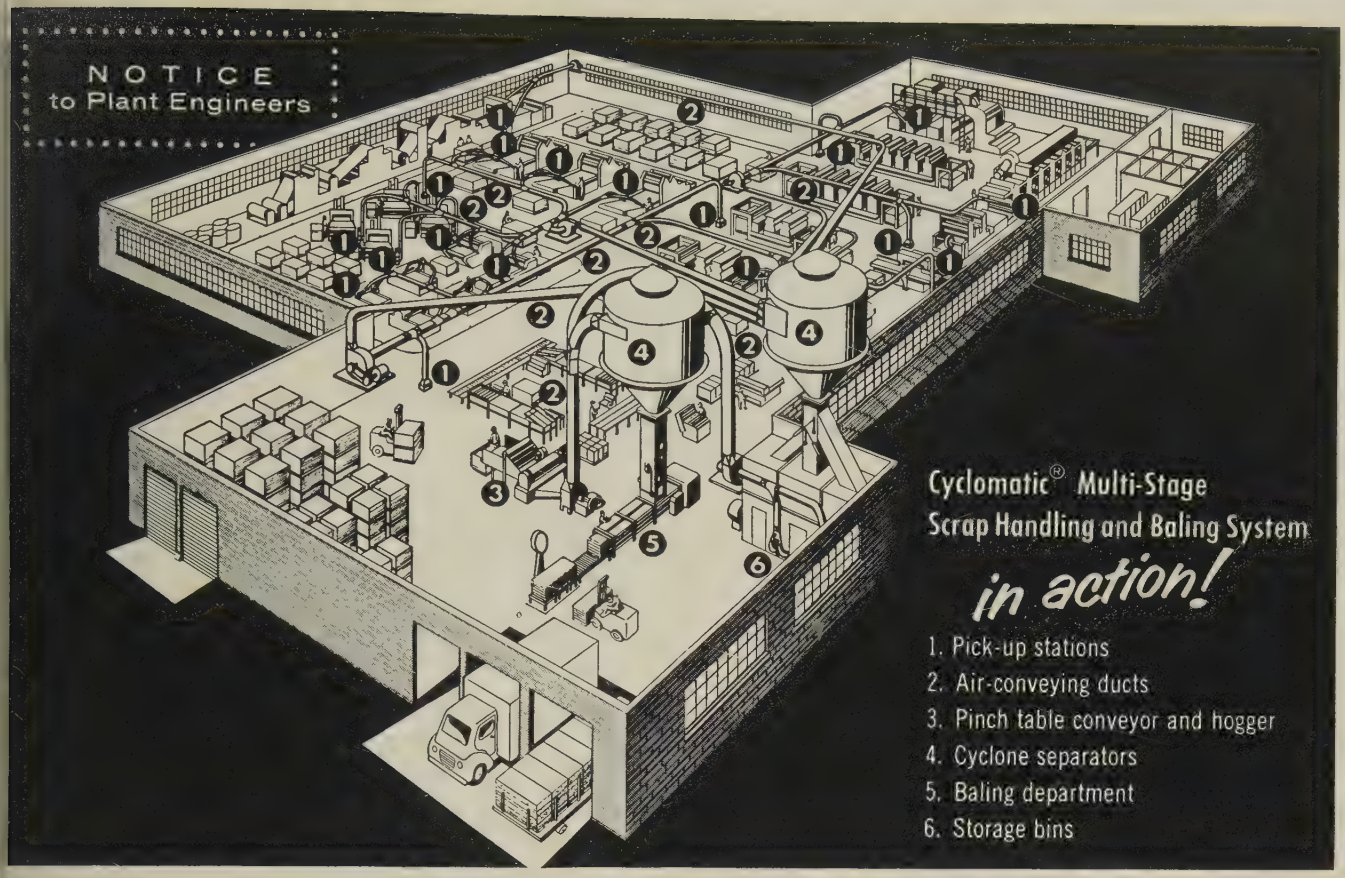

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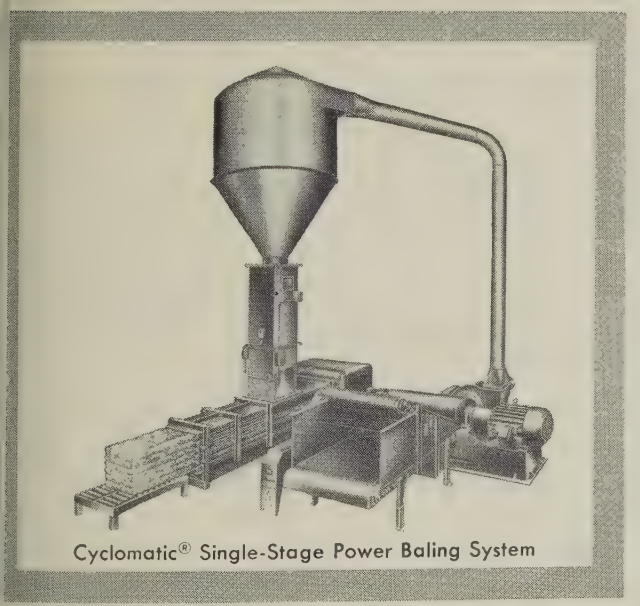

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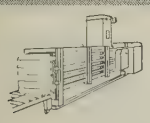

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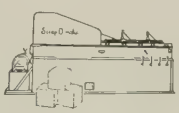

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TAPPI Annual Meeting Program

Commodore Hotel, New York, N. Y., Feb. 22–25, 1960

Forty-Fifth Annual Meeting

The 45th Annual Meeting of the Technical Association of the Pulp and Paper Industry will be held at the Commodore Hotel, New York, N. Y., on Feb. 22–25, 1960.

The following is the tentative program for the Annual Meeting and is subject to further change:

Committee Meetings

Sunday, February 21

Executive Committee	10:30 a.m.	Room A
Joint Advisory Council	9:00 a.m.	Room B
Alkaline Pulping Committee	2:00 p.m.	Room C
Coating Council	2:00 p.m.	Room E
Engineering Data Committee	3:00 p.m.	Room F
Testing Division Steering Committee	2:00 p.m.	Room G

Monday, February 22

Container Testing Committee	9:00 a.m.	
Fibrous Materials Testing Committee	9:00 a.m.	
Fillers & Pigments Testing Committee	9:00 a.m.	
Packaging Materials Testing Committee	9:00 a.m.	
Paper Shipping Sack Testing Committee	9:00 a.m.	
Paper Testing Committee	9:00 a.m.	
Paperboard Testing Committee	9:00 a.m.	
Pulp Purification Committee	10:00 a.m.	East Ballroom Parlor
Pulp Testing Committee	10:00 a.m.	
Pulping, Bleaching and Papermaking Chemicals Testing Committee	9:00 a.m.	
Statistics Committee	9:15 a.m.	Room A
Testing Division Officers	7:30 a.m.	South Room
Technical Operations Committee	7:30 a.m.	Room G
Wax Testing Committee	9:00 a.m.	East Room, Roosevelt
Acid and Semichemical Pulping Committees	2:00 p.m.	
Agricultural Fibers Steering Committee	2:00 p.m.	
Chemical Methods Committee	2:00 p.m.	East Ballroom
Coating Committee	2:00 p.m.	Room B
Corrugated Containers Production Committee	2:00 p.m.	
Corrugated Containers Steering and Advisory Committee	2:00 p.m.	
Cylinder Board Committee	2:00 p.m.	Room E
Deinking Committee	2:00 p.m.	Room A
Dissolving Pulps Committee	2:00 p.m.	
Engineering Division Officers	2:30 p.m.	Hq. Conference Room, 360 Lexington Ave.
Engineering SAC	1:30 p.m.	Hq. Conference Room, 360 Lexington Ave.
Fourdrinier Committee	2:00 p.m.	
Fundamental Research Committee	2:00 p.m.	Room F
Graphic Arts Committee	2:00 p.m.	East Ballroom Parlor
Mechanical Engineering Committee	12:30 p.m.	
Mechanical Pulping Committee	2:00 p.m.	
Microbiological Committee	2:00 p.m.	
Microscopy Committee	2:00 p.m.	West Ballroom
Optical Methods Committee	2:00 p.m.	West Ballroom
Physical Testing Committee	2:00 p.m.	East Ballroom
Plastics Committee	2:00 p.m.	Room C
Plastics Laminates Committee	2:00 p.m.	
Precision Committee	2:00 p.m.	West Ballroom
Routine Control Methods	12:15 p.m.	
Structural Fibrous Materials Committee	2:00 p.m.	Room G
Wax Testing Committee	2:00 p.m.	East Room, Roosevelt

Tuesday, February 23

Chemical Engineering Steering Committee	10:00 a.m.	Room F
Chemical Products Committee	9:00 a.m.	
Corrugated Containers Engineering Committee	9:00 a.m.	
Corrugated Containers Industrial Engineering Committee	9:00 a.m.	
Corrugated Containers Process & Quality Control Committee	9:00 a.m.	
Corrugated Containers Production Committee	9:00 a.m.	
Corrugated Containers Raw Materials Committee	9:00 a.m.	
Forest Biology Committee	9:00 a.m.	Room E
Structural Fibrous Materials Committee	9:00 a.m.	Room G
Wax Testing Committee	9:00 a.m.	East Room, Roosevelt
Beater Additives Subcommittee	2:00 p.m.	East Ballroom Parlor
Chemical Engineering Committee	12:15 p.m.	Room F
Chemical Methods Committee	2:00 p.m.	Room F
Civil Engineering Committee	12:15 p.m.	
Drying Committee	12:15 p.m.	
Electrical Engineering Steering Committee	2:00 p.m.	
Joint Advisory Committee on Printability Research	2:00 p.m.	Room E
Operations Research Subcommittee	2:00 p.m.	Hq. Conference Room, 360 Lexington Ave.
Paper Coating Additives Subcommittee (Project 303)	2:00 p.m.	
Sampling Group, ASTM-TAPPI Task Group	2:00 p.m.	Room 1004
Training & Education Committee	2:00 p.m.	Room G
Wax Testing Committee	2:00 p.m.	East Room, Roosevelt

Wednesday, February 24

Agricultural Fibers Committee	9:00 a.m.	Room A
Converting and Consuming Div. Officers	9:00 a.m.	Room E
Engineering Economics Committee	9:00 a.m.	Room F
Wax Testing Committee	9:00 a.m.	East Room, Roosevelt
Corrosion Committee	12:15 p.m.	Room A
Corrosion Committee	2:00 p.m.	Room A
Fluid Mechanics Committee	12:15 p.m.	Room E
Fourdrinier Committee	2:00 p.m.	Room E
Local Section Operations Committee	4:00 p.m.	East Ballroom Parlor
Wax Testing Committee	2:00 p.m.	East Room, Roosevelt

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Thursday, February 25

Chemical Engineering Problems Subcommittee	9:00 a.m.	Room E
Technical Operations Committee	9:00 a.m.	Room G
Wet Strength & Interfiber Bonding Committee	9:00 a.m.	Room A

MONDAY, FEBRUARY 22, 1960

9:00 a.m. General Session I (West Ball Room)

1. "Deinking—Present and Future," by A. J. Felton, Shartle Division, The Black-Clawson Co., Middletown, Ohio.
2. "The Electrical Properties of Select Fractions of Cellulose Pulps," by T. D. Callinan, J. S. Crimi, P. M. Schwartz, and L. H. Wirtz, International Business Machines Corp., Yorktown Heights, N. Y.
3. "The Application of a Centrifugal Water Retention Test to Pulp Evaluation," by E. F. Thode, J. F. Bergami, Jr., and R. E. Unson, The Institution of Paper Chemistry, Appleton, Wis.
4. "Production of High Wet Strength Paper by Wet End Addition of Dialdehyde Starch," by B. T. Hofreiter, G. E. Stammerstrand, C. L. Mehltretter, W. E. Schulze, and A. J. Ernst, Northern Regional Research Laboratory, USDA, Peoria, Ill.

9:00 a.m. Alkaline Pulping Session (Room B-C)

R. R. Fuller, Gulf States Paper Co., Tuscalusa, Ala., *Moderator*

1. "Comparison Study of Full Chemical Pulping—Alkaline Processes," by J. R. Peckham and V. Van Drunen, Institute of Paper Chemistry, Appleton, Wis.
2. "Cooking of Sawdust by the Sulfate Process," by K. Lauer, University of Alabama, University, Ala.
3. "Commercial Pulping by Total Chemical Using Elemental Sulfur in the Digester," by B. Cox, H. M. Owen, and R. R. Fuller, Gulf States Paper Co., Tuscalusa, Ala.
4. "Effect of Sulfite/Carbonate Ratio on Neutral Sulfite Semi-chemical Pulping," by C. B. Christiansen, Diamond Alkali Co., Painesville, Ohio.

10:30 a.m. Annual Business Meeting (East Ball Room)

J. R. Lientz, Union Bag-Camp Paper Corp., Savannah, Ga., *Chairman*

Secretary-Treasurer's Report
President's Address
Report of Tellers' Committee
Reading of Proposed Changes in the Constitution
Vote on Proposed Changes

2:00 p.m. Chemical Products Session (South Room)

S. Coppick, Scott Paper Co., Chester, Pa., *Chairman*

1. "Uses of Sulfate Turpentine," by C. E. Tyler, Hercules Powder Co., Brunswick, Ga.
2. "Para-hydroxy Benzoic Acid and Other Chemical Products from Aspen Pulping Spent Liquors," by I. A. Pearl, Institute of Paper Chemistry, Appleton, Wis.
3. "Production of Lignin and Organic Acids from Kraft Pulping Liquors," by M. E. Findley, Alabama Polytechnic Institute, Auburn, Ala.

TUESDAY, FEBRUARY 23, 1960

9:00 a.m. Fourdrinier Session (Grand Ballroom)

W. H. Kennedy, Black-Clawson Co., Watertown, N. Y., *Chairman*

1. "Rope Carrier Practices," by H. H. Mullins, Forest Products Div., Olin Mathieson Chemical Corp., West Monroe, La.
2. "A Practical Approach to Open Side Calender Design," by J. E. Perkins, The Black Clawson Co., Watertown, N. Y.
3. The Hydromechanics of Pulp Slurries," by W. L. Ingmanson, The Institute of Paper Chemistry, Appleton, Wis.

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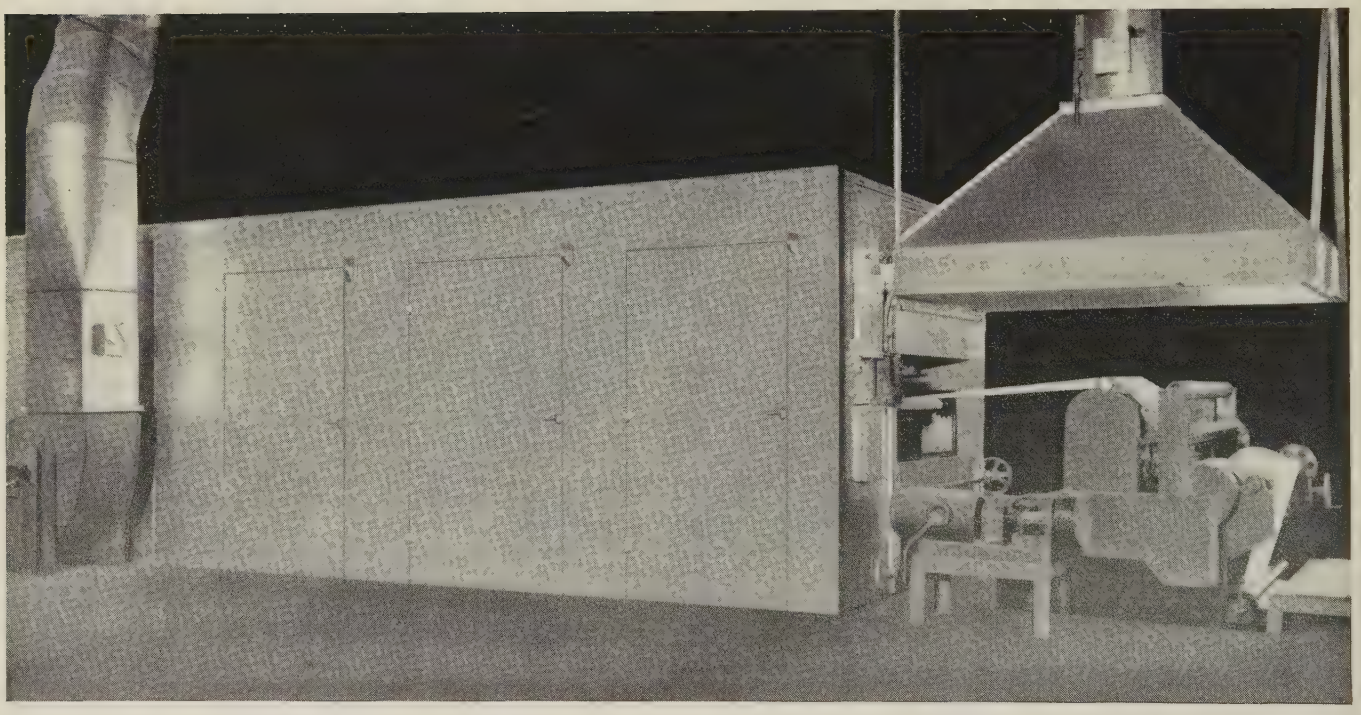

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4. "Process Control as Related to Fourdrinier Operations," by R. L. Traxler, Scott Paper Co., Chester, Pa.

9:00 a.m. Acid Pulping Session I (East Ball Room)

"Sulfite Pulping Recovery Systems," P. J. Frost, Crown Zellerbach Corp., *Chairman*, J. L. McCarthy, University of Washington, Seattle, Wash., *Moderator*

1. "Magnefite and Magnesium Base Recovery," by G. H. Tomlinson II, Howard Smith Paper Mills, Cornwall, Ont., Canada.
2. "Atomized Suspension Technique," by W. H. Gauvin, Pulp and Paper Research Institute, Montreal, Que., Canada.
3. "Sivola Process," by E. H. Kennedy, Combustion Engineering, Inc., New York, N. Y.

9:00 a.m. Cylinder Board Session I (West Ball Room)

G. T. Renegar, Container Corporation of America, Philadelphia Pa., *Chairman*

1. "Application of Wires in the Paperboard Industry," by N. O. Weil, W. S. Tyler Co., Cleveland, Ohio.
2. "The Practical Aspects of Slime Control in the Board Mills," by J. J. Dietzel, Buckman Laboratories, Inc., Memphis, Tenn.
3. "Chemical Cleaning Procedures in Paperboard Industry," by R. Kipp, Oakite Products, Inc., New York, N. Y.
4. "Whys and Hows of Cleaning Paper Stock for Board Mills," by D. F. Lehman, Black-Clawson Co., Shartle Div., Middletown, Ohio.

9:00 a.m. Adhesives Testing Session (Room B-C)

W. W. Sederlund, National Starch & Chemical Co., Plainfield, N. J., *Chairman*

1. "A Measure of Surface Tension and Surface Film Elongation," by P. B. Davidson, Mellon Institute, Pittsburgh, Pa.
2. "Practical Application of the T-A-C Tester," by W. Neuse, Armour & Co., Alliance, Ohio.
3. "The Theory of Adhesion," by J. J. Bikerman, Massachusetts Insitute of Technology, Cambridge, Mass.

9:00 a.m. Agricultural Fibers Session (Room A)

F. B. Grunwald, Consolidated Paper Co., Monroe, Mich., *Chairman*

1. "A Search for New Fiber Crops," by H. J. Nieschlag, G. H. Nelson, I. A. Wolff, and R. E. Perdue, Jr., Northern Utilization Research and Development Div., USDA, Peoria, Ill.
2. "Water Sorption of the Main Plant Cell Wall Constituents," by M. F. Luthgens, Covington, Va.
3. "Microscopic Investigations of the Morphology of Pulp Fibers," by K. A. Jurbergs, Buckeye Cellulose Corp., Memphis, Tenn.
4. "Continuous Pulping Experiments," by C. J. Wynstra, Central Fibre Products Div., Packaging Corp. of America, Quincy, Ill.

2:00 p.m. Water Session I (South Room)

O. F. Hutchinson, Peter Cooper Corp., Gowanda, N. Y., *Chairman*

1. "Design of Paper Machine Wet Ends for Maximum Internal Water Use in a Paper Mill," by H. L. Berg, Sveen-Pedersen Sales Corp., Long Island City, N. Y.
2. "Operating Functions from Efficient Wet End Design of a Paper Machine," by O. F. Hutchinson, Peter Cooper Corp., Gowanda, N. Y.
3. "Water Use Panel":
   Fresh Water Showers: A. E. Gray, A. E. Broughton Co., Glens Falls, N. Y.
   White Water Showers: (Speaker to be announced)
   Savealls: H. Nelson, Hawthorne Paper Co., Kalamazoo, Mich.
   Retention Aids: D. Clark, Simpson Paper Co., Everett, Wash.
   Bactericides: M. Schirtzinger, Mead Corp., Chillicothe, Ohio
4. Discussion

2:00 p.m. Corrugated Containers Session I (Grand Ball Room)

C. H. Robson, Lanzit Corrugated Box Co., Chicago, Ill., *Moderator*

1. "Supervisory Practices in Container Plants," by R. W. Buttery, Bathurst Containers, Ltd., Montreal, Canada.

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2. "Unitizing of Corrugated Containers," by J. Comly, Connelly Containers, Inc., Philadelphia, Pa.
3. "Operating Experiences with Tapered Mechanical Glue Roll Drives," by W. Salogga, Corrugating Technicians, Inc., Tenafly, N. J.
4. "Scheduling a Container Plant," by R. P. Caponi, Union Bag-Camp Paper Corp., Chicago, Ill.

2:00 p.m. Acid Pulping Session II (East Ball Room)

J. L. McCarthy, University of Washington, Seattle, Wash., *Moderator*

1. "Sodium-Base Recovery—Western Precipitation Process," by R. Q. Boyer, Western Precipitation Corp., Los Angeles, Calif.
2. "Sodium-Base Recovery—The Mead Process," by W. J. Darmstadt, Babcock and Wilcox Corp., Barberton, Ohio.
3. "Sodium-Base Recovery—Stora Kopperburg Process," by K. N. Cedarquist, K. Ahlborg, B. O. Lunden, Stora Kopperbergs, Bergslags AB, Falun, Sweden, and T. O. Wentworth, Chemical Processes, Inc., Cleveland, Ohio.

2:00 p.m. Cylinder Board Session II (West Ball Room)

G. T. Renegar, Container Corporation of America, Philadelphia, Pa., *Chairman*

1. "Rubber Covered Rolls on Cylinder Board Machines," by J. J. Conner, Raybestos-Manhattan, Inc., Passaic, N. J.
2. Discussion of Papers.

2:00 p.m. Microbiological Session (Room B-C)

W. M. Van Horne, The Institute of Paper Chemistry, Appleton, Wis., *Chairman*

1. "The Correlation and Precision of 5-Day and Manometric Biochemical Oxygen Demand in the Range of 1 to 20 Parts Per Million," by E. Dillingham, The Institute of Paper Chemistry, Appleton, Wis.
2. "A Research Tool Adapted for Use in the Industrial Laboratory," by L. L. Wolfson, Peter Song, and J. R. Nelson, Nalco Chemical Co., Chicago, Ill.
3. "A New Candidate Slimicide," by M. Legator, Shell Development Co., and A. N. Stern, West Virginia Pulp and Paper Co., Covington, Va.
4. Panel Discussion—A. N. Stern, *Chairman*
   "The Position of Toxicants Under New Food and Drug Amendments."
   Participants: A. N. Stern, West Virginia Pulp and Paper Co., Covington, Va., B. F. Shema, Betz Laboratories, Inc., Philadelphia, Pa., and L. Hazleton, Hazleton Laboratories.

2:00 p.m. Water Session II (South Room)

R. J. Keating, Grover Water Conditioning Co., New York, N. Y., *Chairman*

1. "Water Treatment in Paper Mill Power Plants," by H. Bacon and L. von Lossberg, Sheppard T. Powell, Baltimore, Md.
2. "Classification of High Color Waters," by C. T. Wise, J. R. Sirrine Co., Greenville, N. C.
3. "Water Treatment at Abitibi Mills," by G. Stewart, Abitibi Power and Paper Co., Toronto, Ont.
4. "Water Conservation by External Reuse," by L. Klinger, Whippany Paperboard Co., Whippany, N. J.

2:00 p.m. Structural Fibrous Materials Session (Room A)

R. S. Von Hazmburg, United States Gypsum Co., Chicago, Ill., *Chairman*

1. "Factors Affecting the Warping of Fiberboard Roof Insulation," by J. J. Perot, Jr., National Gypsum Co., Buffalo, N. Y.
2. "Dielectric Heating for Drying Wood Fiberboard," by D. C. Carter, Johns-Manville Corp., Manville, N. J.
3. "Unexpected Variables Affecting the TAPPI-SFMC Drainage Time Tester of Suggested Method T 1002 sm-51," by W. G. Coggan.
4. "The Relation of Fiber and Pulp Properties of the Properties of Structural Fiberboard Products," by E. J. Jones, The Institute of Paper Chemistry, Appleton, Wis.
5. "Asphalt and Its Use in Insulating Board," by E. M. Lorrenzine, American Bitumuls and Asphalt Co., San Francisco, Calif.

WEDNESDAY, FEBRUARY 24, 1960

9:00 a.m. Corrugated Containers Session II (Grand Ball Room)

G. W. Van der Ploeg, Hoerner Boxes, Inc., Keokuk, Iowa, *Moderator*

1. "Product Development and the Production Merry-Go-Round," by H. J. Shlotter, Hoerner Boxes, Inc., Keokuk, Iowa.
2. "New Methods for Production Control of Silica Non-Slip Applications," by H. J. Wilhoyte (Joint survey by du Pont, Cornell, and Ortman-McCain).
3. "Production, Progress, and the Manufacturers Joint," by J. J. Kipnees, Container Laboratories, Inc., New York, N. Y.
4. "Sources of Errors and Practices Contributing to Their Reduction," by W. J. Hurrell, Hinde & Dauch, Toronto, Canada.

9:00 a.m. Graphic Arts Session I (East Ball Room)

J. H. Wing, Continental Can Co., Uncasville, Conn., *Chairman*

1. "Factors Affecting Gloss Hold-Out," by J. M. Fetsko and A. C. Zettlemoyer, Lehigh University, Bethlehem, Pa.
2. "Photomicrography of Paper Surfaces and Their Printing Characteristics," by I. M. Bernstein, Flexo-Inks, Inc., Newark, N. J.
3. "The NPIRI Printing Wedge as a Measure of Commercial Printability of Newsprint," by S. Steinberg, New York Times, New York, N. Y., and C. F. Geffken, and W. K. Herrmann, J. M. Huber Corp., New York, N. Y.
4. "Predicting Printing Quality," by A. Glassman, R. R. Donnelley & Sons Co., Chicago, Ill.

9:00 a.m. Mechanical Pulping Session (West Ball Room)

M. S. Htoo, International Paper Co., Glens Falls, N. Y., *Chairman*

1. "New Process for Hardwood Pulping," by H. L. Crosby and N. N. Coe, Dorr-Oliver, Inc., Stamford, Conn.
2. "Low Power Cold Soda Pulping," by E. R. Gremler, The Black-Clawson Co., Hamilton, Ohio.
3. "Steam Cooking of Northern Hardwoods," by D. A. Feigley and M. J. Showalter, Armstrong Cork Co., Lancaster, Pa.
4. "Underwater versus Land Wood Storage and Effect on Groundwood Pulp," by H. Dailey, Diamond National Corp., Ogdensburg, N. Y.
5. Panel Discussion: "Mechanics and Operation of Quality Control Systems in Groundwood Mills": M. Hughson, *Chairman*, F. W. Bishop, J. K. Kirkpatrick, I. McArthur, R. V. Osborne, J. W. Wenger, and A. Yankowski.

9:00 a.m. Dissolving Pulps Session I (Room B-C)

T. L. Kirtley, Hercules Powder Co., Hopewell, Va., *Moderator*

1. "Xanthates Sulfur Determination by the Batch Ion Exchange Method," by L. H. Phifer and J. L. Bell, American Viscose Corp., Marcus Hook, Pa.
2. "Rapid Method for Determination of Total Sulfur in Viscose," by J. F. Crow, J. T. Dux, and L. H. Phifer, American Viscose Corp., Marcus Hook, Pa.
3. "Tire Cord Fatigue in Fleet Tested Tires," by W. G. Klein, Fabric Research Laboratories, Dedham, Mass.
4. "Structure Changes in Alkali Cellulose," by K. H. Lauer, University of Alabama, University, Ala.

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9:00 a.m. Statistics Session I (South Room)

M. S. Renner, Dewey and Almy Chemical Div., W. R. Grace & Co., Cambridge, Mass., *Chairman*

1. "The TAPPI Statistics Committee Attributes Sampling Program," by W. E. Carlson, St. Regis Paper Co., Carthage N. Y.
2. "What Use Is Statistics to Me?," by P. C. Clifford, Montclair State College, Montclair, N. J.
3. "An Application of Operations Research to a Pulp Wood Inventory Control Problem," by T. A. Hewson, St. Regis Paper Co., New York, N. Y.

2:00 p.m. Corrugated Containers Session III (Grand Ball Room)

H. T. Scordas, Union Bag-Camp Paper Corp., New York, N. Y., *Moderator*

Open Forum. Panel Members: All authors

2:00 p.m. Graphic Arts Session II (East Ball Room)

L. E. De Lauter, West Virginia Pulp & Paper Co., Chicago, Ill., *Chairman*

1. Panel: "Paper from the Printer's Viewpoint." A panel of qualified people from various phases of the printing industry will discuss paper as they view it from their industry. Each speaker will present a short talk on his particular phase of printing. A question and answer period will follow the talks.

Flexography: I. M. Bernstein, Flexo-Gravure Inks, Inc., Newark, N. J.

Gravure: H. Smock, Chicago Rotoprint Co., Chicago, Ill.

Letterpress: F. B. Lincoln, Jr., Time, Inc., New York, N. Y.

Lithography: A. Donaldson, Jr., Strobridge Lithographing Co., Cincinnati, Ohio.

Silk Screen: V. Strauss, New York, N. Y.

2:00 p.m. Pulp Purification Session I (West Ball Room)

F. Kraft, Marathon Div., American Can Co., Menasha, Wis., *Chairman*

1. "A Study of the Hot Alkaline Degradation of Carbohydrates by Means of U. V. Absorption," by D. Rivington and H. Kraessig, Industrial Cellulose Research, Ltd.
2. "Hypochlorite Bleaching of Dissolving Pulps," by W. L. Thornburg and J. W. Smith, Buckeye Cellulose Corp., Memphis, Tenn.
3. "Demethylation of Aromatic Methylethers and Its Relation to Chlorination of Lignin and Wood Pulps," by K. V. Sarkanen and R. W. Strauss, College of Forestry, Syracuse, N. Y.

2:00 p.m. Dissolving Pulps Session II (Room B-C)

M. Plunguian, Hercules Powder Co., Wilmington, Del., *Moderator*

1. "The Oxidation of the ICCA Pulps with Lead Tetra-acetate," by R. W. Detrick, Thilmany Pulp and Paper Co., Kaukauna, Wis.
2. "Chemical and Physical Nature of Insolubles in Acetone Solutions of Cellulose Acetate," by M. Easterwood and L. H. Sperling, Buckeye Cellulose Corp., Memphis, Tenn.
3. "Dissolving Pulps from Domestic Timber Bamboo—*Phyllostachys bambusoides*," by T. R. Naffziger, R. S. Matuszewski, T. F. Clark, and I. A. Wolff., Northern Utilization Research Div., USDA, Peoria, Ill.

2:00 p.m. Statistics Session II (South Room)

W. E. Carlson, St. Regis Paper Co., Carthage, N. Y., *Chairman*

1. "Some Underlying Considerations in Estimating Basis Weight and Basis Weight Variation," by M. S. Renner, Dewey and Almy Chemical Div., W. R. Grace & Co., Cambridge, Mass.
2. "Basis Weight Variation from the Consumer's Point of View," by D. Looney, Polaroid Corp., Cambridge, Mass.
3. "Basis Weight Variation and Control from the Supplier's Point of View," by F. Hartwell, Peter J. Schweitzer, Inc., Lee, Mass.
4. "A Statistically Based Sampling Procedure for Estimating Basis Weight," by J. H. Moyer, The Champion Paper and Fibre Co., Texas Div., Pasadena, Texas.

THURSDAY, FEBRUARY 25, 1960

9:00 a.m. Dissolving Pulps Session III (East Ball Room)

M. Heath, Buckeye Cellulose Corp., Memphis, Tenn., *Moderator*

1. "Electron Microscopical Studies of Alterations of Fiber Structure Effected by Swelling" by G. Jayme and G. Hunger, Institut für Cellulosechemie, Darmstadt, Germany.
2. "Phenomena during Swelling and Dissolving of Fibrous Plant Cells in New Cellulose Solvents" by G. Jayme and M Harders-Steinhäuser, Institut für Cellulosechemie, Darmstadt, Germany, [Film—commentary by G. Hunger]

9:00 a.m. Pulp Purification Session II (West Fall Room)

F. Kraft, Marathon Div. American Can Co., Menasha, Wis., *Chairman*

1. "Oxidation Potential for Control in the Bleach Plant," by A. D. Harbin, Jr., Diamond Alkali Co., Painesville, Ohio.
2. "Use of Titanium in Pulp Bleaching Equipment," by G. W. Bauer, Mallory Sharon Metals Corp., Niles, Ohio.
3. "Refiner Bleaching of Cold Caustic Pulp," by F. Jahne, Diamond National Corp., Ogdensburg, N. Y.
4. "A Review of Continuous Production of Hypochlorite in the Paper Industry," by R. S. Ericson and I. C. MacGugan, Wyandotte Chemicals Co., Wyandotte, Mich.

9:00 a.m. Corrosion Session (Room B-C)

E. W. Hopper, Pittsburgh, Pa., *Chairman*

1. "Overlay Installation and Corrosion in Alkaline Digesters," by D. Innes, International Paper Co., Mobile, Ala., L. Dela Grange, West Virginia Pulp and Paper Co., New York, N. Y., and M. Cyr, Fraser Co. Ltd., Newcastle, N. B.
2. "Neutral Sulfite Pulping Corrosion Problems," by E. K. Scholz, West Virginia Pulp and Paper Co., Covington, Va., and L. H. Park, Union Bag-Camp Paper Corp., Savannah, Ga.
3. "Alkaline Pulping Corrosion Problems," by W. E. Hendricks, Mead Corp., Chillicothe, Ohio, and W. C. Barnwell, Gaylord Container Div., Crown Zellerbach Corp., Bogalusa, La.
4. "Costs of Corrosion," by W. Muller, PPRIC, Montreal, Que., and W. K. Boyd, Battelle Memorial Institute, Columbus, Ohio.
5. "The Influence of Composition Balance and Control on Cast Stainless Alloys Designed for the Pulp and Paper Industry," by J. M. Wilcox, W. R. Barber, Jr., and D. D. Burgan., Electric Steel Foundry Co., Portland, Ore.

9:00 a.m. General Session II (South Room)

1. "Dispersion of Asphalt Materials in Paper Stocks by the Petro-Diffusion System," by L. Van Guelpen, The Black-Clawson Co., Middletown, Ohio.
2. "An Integrated Nitric Acid Pulping Process," by D. L. Brink, University of California, Richmond, Calif.
3. "Contour Control of Paper Calenders," by M. D. Stone and A. T. Liebert, United Engineering and Foundry Co., Pittsburgh, Pa.

9:00 a.m. Drying Session (Grand Ball Room)

1. "An Optimized High Velocity Air Drier," by R. Hurm, Beloit Iron Works, Beloit, Wis.
2. "Heat Transfer and Water Removal in Cylinder—Part I Unfelted Cylinders," by A. H. Nissan and D. Hansen, Rensselaer Polytechnic Institute, Troy, N. Y.
3. "Low Temperature Radiant Heat Drying," by H. L. Smith, Horace L. Smith, Jr., Inc., Richmond, Va.

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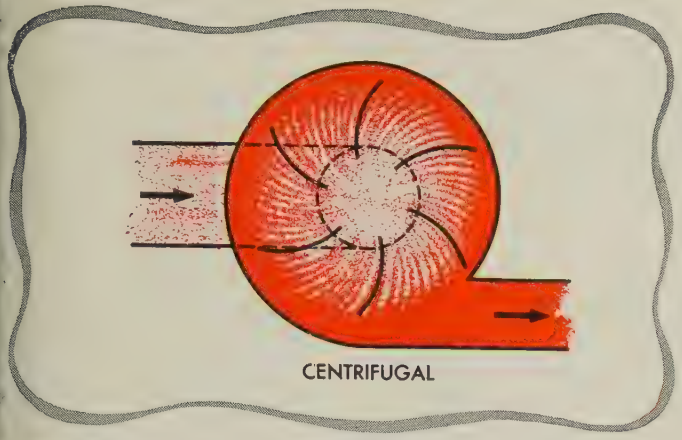

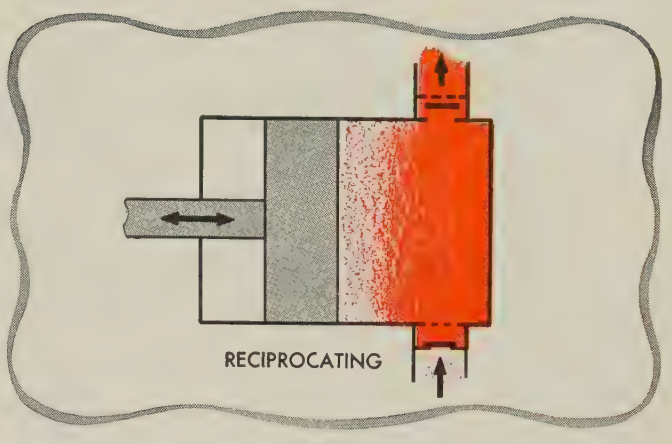

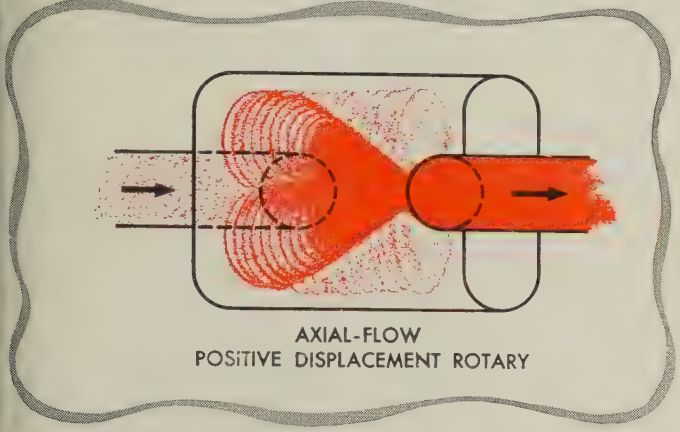

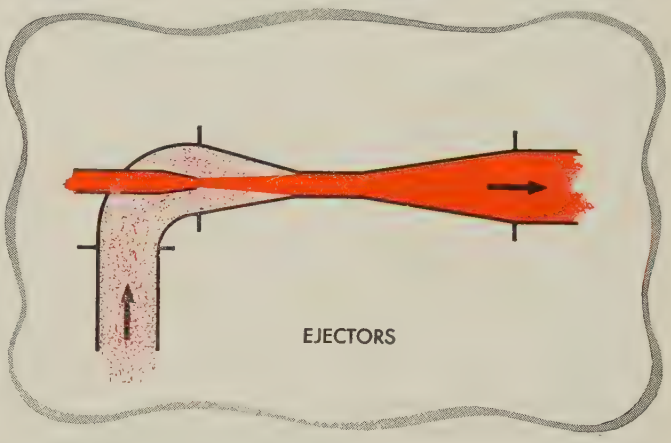

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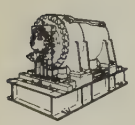
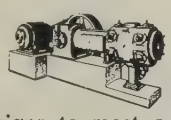
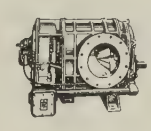


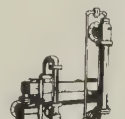
Centrifugal Compressors. Ingersoll-Rand centrifugal blowers and compressors are available in single- and multi-stage units, for handling large volumes of air and all other gases at vacuum and for pressures up to 2500 psi and higher. Sizes range up to 20,000 hp and capacities to 300,000 cfm. They can be driven by steam turbine, electric motor, combustion-gas turbine and hot-gas expander turbine.



Reciprocating Compressors. I-R reciprocating compressors are available in single- and multi-cylinder designs to meet a wide range of high-pressure, low-volume requirements. They have been furnished in sizes from ½ to 7,500 hp, for pressures to 35,000 psi and vacuums. Driver types may be electric, steam, oil or gas, and oil-free air can be provided by means of Type NL non-lubricated cylinders and valves.



Axi-Compressors. The Ingersoll-Rand Axi-Compressor is a compact, space-saving axial-flow positive displacement rotary compressor that offers unique advantages within its operating range. It delivers a steady flow of clean, oil-free air or gas and can be direct driven by electric motors or gasoline engines. Available in capacities from 100 to 12,000 cfm for pressures to 15 psi and vacuums to 15″ hg. Higher vacuums can be produced with water injection or with two stage units.



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Forecast of 1960 Paper Production

LOUIS T. STEVENSON

We summarize our forecast for the year 1960 and our revised estimate for 1959 in the following table:

Paper Production

	Million tons	
	Forecast 1960	*Revised estimate 1959*
Paper (ex building and newsprint)	13.3	12.9
Paperboard (ex building board)	16.5	15.8
Newsprint	2.0	1.9
Building paper and board	3.5	3.4
Total	35.3	34.0

THE 1959 EXPANSION

As expected, the year 1959 has been a period of great expansion in the volume of paper produced. The economy as a whole picked up rapidly in the first six months, or until the steel strike, with the Federal Reserve Board adjusted index of industrial production rising 8% from January to June. Since then the steel strike has had a cumulatively damaging effect on the economy, with a drop in the index of 5% through October.

Figure 1 shows the effect of steel strikes on both the Federal Reserve Board durable goods and nondurable goods production indexes in the period from January, 1956, through October, 1959. In order to minimize the difference in growth rates between the several series, each of the indexes has been converted to a 1956–58 base. The index of paper production is a 4-month moving average centered on the third month. A series of these three on a 1947–49 base shows that the paper production index, which is included in the nondurable index, has grown more rapidly than nondurables but fluctuates much less than the durable goods index.

Paper production is more closely related to nondurables than to durables. Nondurables, except in major recessions, do not follow the movement of durable goods. That index in turn is responsive to changes in steel production. However, although steel strikes have had a radical effect on durables, it is clear that up to the moment they have had little or no effect on the production of paper and board. What lies in the future is uncertain because this strike has been the longest on record and, if it should be resumed after the 80-day cooling off period, it might result in throwing the economy into a temporary recession. It is not expected that this will happen, and by January, 1960, when the period for this forecast starts, this situation should be clarified. As we write this in November, 1959, we are assuming that the steel strike will have been settled in January. In that case a rebound of durable goods production and the economy as a whole is expected to carry the present industrial production index above its June, 1959, high. This is another way of saying that the durable goods segment of the economy will recover its balanced relationship with the nondurables and that the interrupted upswing will be resumed.

The high interest rates which we expect will prevail are likely to dampen new construction activity and prevent the runaway boom which might otherwise occur.

The year 1959, with a $10^1/_2\%$ increase in paper and board production over 1958, appears to be a boom year. But is it? Can it be called a boom if the production has returned just to its normal growth pattern after a recession? To be sure, the 1959 rise has been sharper than we expected, but we still maintain that it has not reached boom proportions until it moves well above the normal growth line.

Our forecast for 1959 indicated a production volume of 33 million tons, 6.9% above 1958, or 3 percentage points higher than the 3.9% average growth rate. But we also said, "The fact that 1957 and 1958 production was below trend during the recent recession indicates that, with recovery well started, production volume should swing back to its normal growth line. Although the many increases in the use of paper that will be necessary to bring this about are impossible to evaluate, we feel confident that the long term growth pattern will continue at substantially the same rate. If it should, our 1959 forecast, which is a trifle below the trend line, may prove to be low." As a matter of fact, our forecast was about 3% low and the estimated 1959 production is just a shade above the trend line as shown in Fig. 2. We also indicate our projected 1960 production volume on this same chart. It will be observed that our projection for 1960 is slightly above the trend line. This we believe is a reasonable reaction to expect from the depressed period of 1957–58.

INDUSTRY GROWTH RATE

The persistence of this strong growth trend is quite

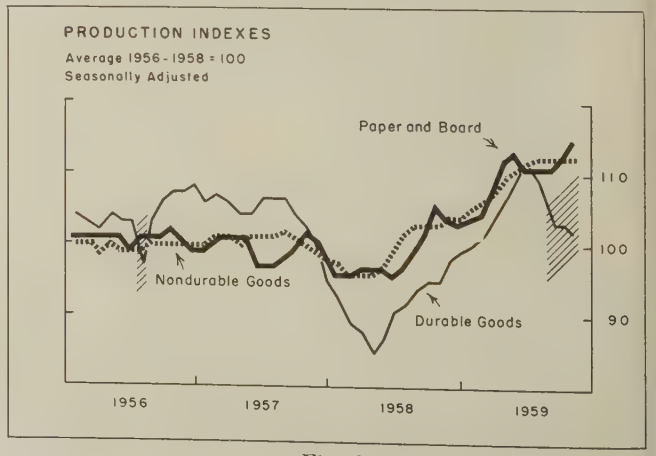

Fig. 1

Louis T. Stevenson, Economist, Tucker, Anthony and R. L. Day, 120 Broadway, New York 5, N. Y.

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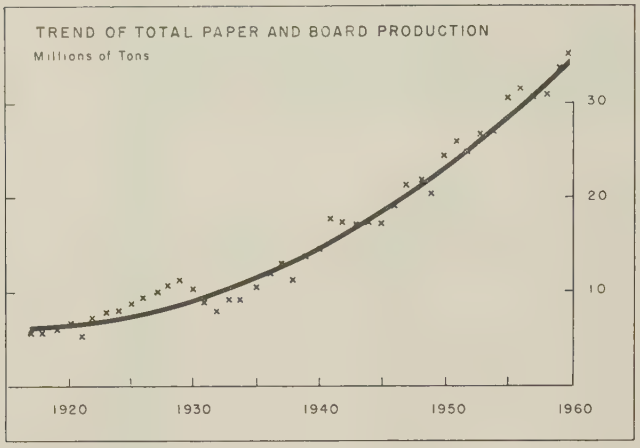

Fig. 2

remarkable for such an old industry which started in America in Colonial days. The industry through its research and development, is constantly developing new techniques, new raw material sources, new products, and new applications of the papermaking processes to multitudes of new uses. The resultant steady growth of 3.9% cumulatively applied on a compound interest basis is apparent in the remarkably steady growth curve shown in Fig. 2. This is of paramount importance in an appraisal and forecast of the industry's prospects. Fluctuations are to be expected but for many years dislocations due to wars and cyclical swings have been small when related to the total volume.

PAPERBOARD FORECAST

In our paperboard forecast we are this year using the grade terminology of the National Paperboard Association instead of that used by the Bureau of the Census. Inasmuch as we have recently covered the subject in our brochure "The Paper Industry Inventory Position," we will not go into detail with respect to inventories. We believe that up to now there has been, broadly speaking, no inventory building. On this basis we forecast 1960 paperboard production by major segments for the year 1960 as follows:

1960 Forecast—Paperboard Production

	1000 tons	
Containerboard		
Liner	5650	
Corrugating medium	2600	
Chip and filler	250	
Total containerboard		8,500
Boxboard		
Folding	2900	
Setup	600	
Special food	1600	
Other	2900	
Total boxboard		8,000
Total paperboard		16,500

PAPER PRODUCTION 1960

We expect the year 1960 will start off with reduced inventories in the following group.

The improvement in general business conditions subsequent to a settlement of the steel strike is likely to result in an increased volume of paper consumption in these grades. This year we are grouping printing and fine papers together because the same machine can be used for making either. There is a possibility that a build-up of inventories may take place and thus raise the production volume above our forecast which follows:

1960 Forecast—Paper Production

	1000 tons
Printing and fine paper	6,200
Coarse and special industrial paper	4,900
Tissue and sanitary paper	2,200
Total paper	13,300

NEWSPRINT PRODUCTION IN THE UNITED STATES

In this country newsprint supplies, unlike other grades of paper, are duty free and almost three-fourths of the volume consumed comes from other countries, chiefly Canada. Consequently the volume of United States production depends upon a balancing of demand against the supply factors of United States and foreign producers. The proportion of newsprint demand that is met by United States mills has been steadily growing. But international supply factors do change and may tend to modify our forecast of United States production in 1960 of 2,000,000 tons.

BUILDING PAPER AND BOARD

The year 1959 has been a good one for this segment of the paper industry, the tonnage of which has been boosted by increases in housing starts and new construction. Housing starts, we expect, will be lower in 1960 but we believe that other types of construction are likely to grow so that, on the whole, the volume of building paper and board should rise slightly to 3,500,000 tons.

THE INTERNATIONAL PICTURE

The rapid growth in the consumption of paper in all countries is stimulating production throughout the world. A 1958 world annual capacity of over 81 million tons is expected to rise to over 100 million tons in the period 1962–65. Over half of this 1958 capacity, 45 million tons was in North America and this is expected to rise to more than 49 million tons by 1962, an increase of about 10% in the four years.

The rest of the world outside of North America is expected to increase its capacity by 40% through the year 1965, or by almost 6% a year for the seven years. Even this rapid growth of capacity will leave both North America and the rest of the world in 1965 with a slight deficit of paper and board capacity below projected demand. Only in North America will newsprint capacity be in a slight surplus above demand while the deficits in other grades will bring the totals to a net deficit.*

The major producing areas are North America and Europe. The European production facilities may be divided into two major groups—Scandinavia and Western Europe including Britain. Scandinavia has pulpwood and the pulp manufactured is either integrated to paper mills or marketed to other countries including Britain, France, and the Low Countries. Most of these mills are nonintegrated and buy pulp or other fibers for conversion into paper. Wood pulp is

* Source: FAO Report, Rome, Sept. 14–19, 1959.

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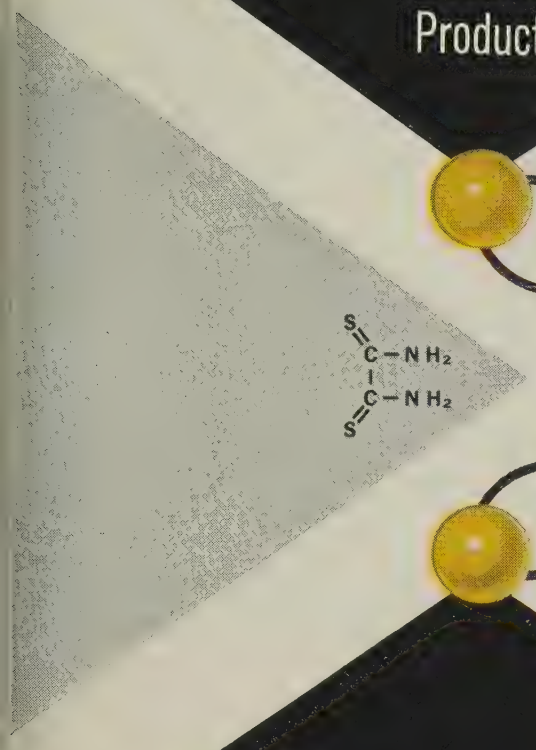

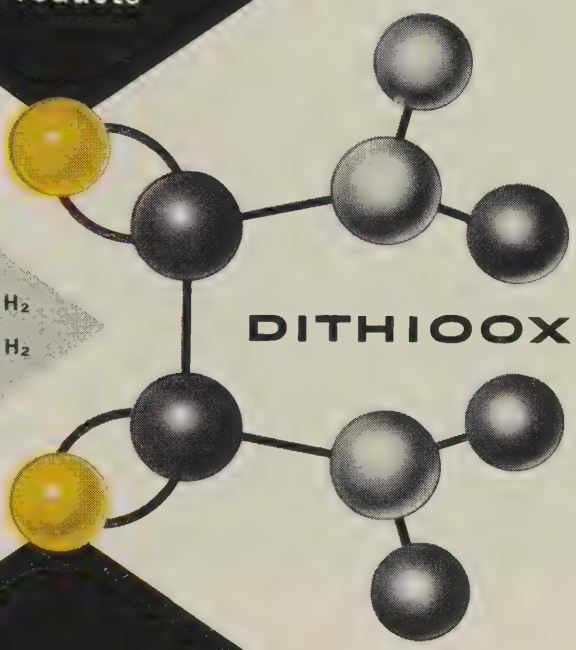

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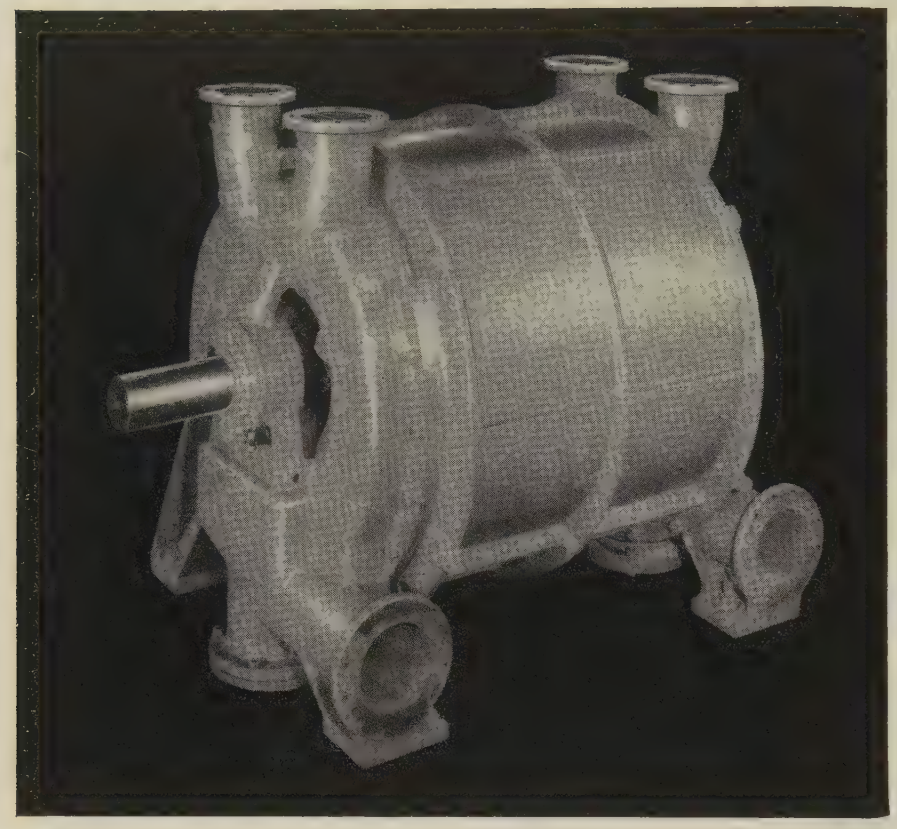
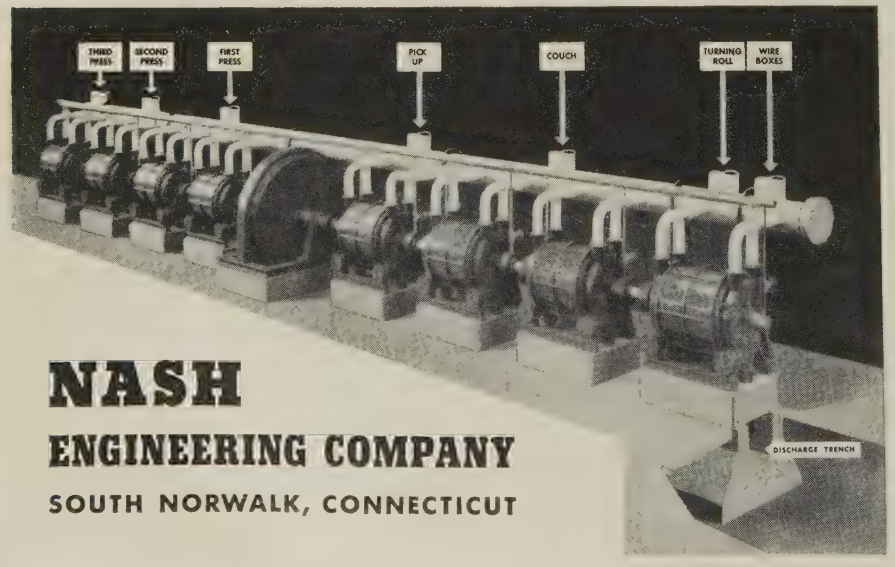


The Nash 5308-A has four separate suction inlets, each of which functions independently of the others. This offers the machine operator great flexibility, since these may be used in any desired combination to produce a variety of capacities and vacuums.

Picture below shows simple drive possible and flexibility of piping permitting capacities to exactly fit requirements of different sections of the paper machine.



NASH
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bought mainly from Scandinavian, Austrian, and North American producers.

The new European Economic Community is, for practical purposes, a "have not" group with respect to wood pulp but if it is joined by the "outer seven" it appears that Scandinavian and Austrian wood pulp and paper will probably be in a preferred position vis-a-vis North American competition with respect to the fast growing European market.

The British paper industry, unless special considerations are stipulated in the treaty, is likely to be in a precarious position, the principal deterrent to British imports of paper from Scandinavia being merely the limitations of the wood supply at the Scandinavian mills. Shortage of wood supply has been no deterrent so far to the considerable expansion of pulp capacity in Scandinavia and it seems quite possible that additional wood could be secured from other areas including Baltic Provinces even though they be under Russian control.

It seems reasonable to expect that Britain will not allow its well-established and competent papermaking industry to be sacrificed. But as yet these problems are not clearly resolved.

Whatever else happens it seems clear that papermakers in the "inner six" (the European Economic Community) are destined to reap most of the benefits from the growth in the European demand for paper. They are expected to be able to operate within a "tariff wall" for their paper products and get their pulp duty free.

If the "inner six" are joined by the "outer seven," Scandinavian producers of paper should also benefit, and Scandinavian market pulp is likely to dominate in its field. Pulp producers in North America may expect at the least the keen competition they have been meeting in Europe to continue indefinitely.

Under the circumstances outlined above, it appears that for American paper companies to benefit from the expected sharp expansion of European paper consumption, they must operate paper mills within the European common market.

Dec. 4, 1959

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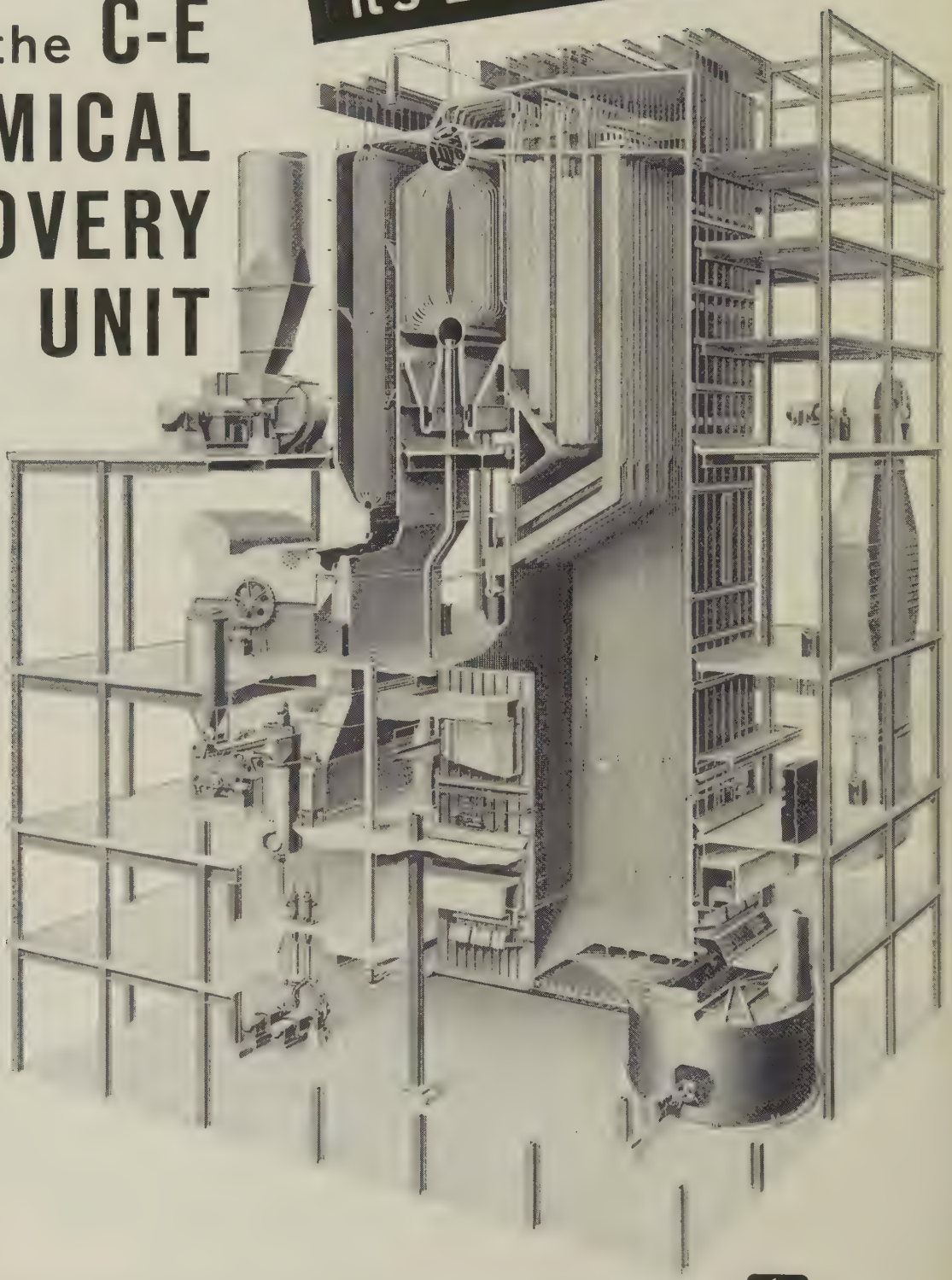

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"STROBE" SHOT SHOWING MOVEMENT OF WATER AFTER LEAVING NIP. PHOTO BY C. B. DAHL

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Food Packaging Papers and the Food Additives Amendment of 1958

ANTHONY W. PESCH

When the Gulf Coast Section chairman, William Elsevier, invited me on behalf of the program chairman to present a paper before the Gulf Coast Section of TAPPI about food packaging papers as affected by the Food Additives Amendment of 1958, Mr. Elsevier suggested that—having "lived" with food packaging paper details very closely for years—I would be able to talk to you extemporaneously for about 30 min, or an hour, without having to write out any formal speech. I accepted Mr. Elsevier's invitation; but this will not be any off-the-cuff talk.

As soon as I began to think about the promised paper at all, three things became clear immediately: first, that the original proposed title, "The FDA Situation," might have implied that I was saying, or thinking, some things about some controversial and/or political matters that I shall try, most scrupulously, to avoid. Second, that I should confine myself as much as possible to food packaging difficulties insofar as papers and paperboards are concerned merely mentioning, at the beginning, that the scientific and technical literature covers the similar difficulties of various of the nonpaper packaging materials adequately enough for most purposes; in fact, my reason for mentioning that some competing packaging industries, also, have some difficulties under the concerned Amendment is because the various troubles of some of those competing materials are sometimes parallel to, or identical with, those of our own industry. If they resolve some of their troubles, it will help us to resolve some of ours. Third—and most important to me, personally—that I should read from a carefully prepared manuscript—i.e., not speak extemporaneously—in order that there would be very little likelihood that I could be misunderstood.

Certain portions of the specifically concerned laws, and certain difficulties connected with complying with said laws, and certain problems of administering them, are so highly technical—both legally and scientifically—that I shall confine this paper to the following: (1) something on the history of Federal legislation in the field of foods, drugs, and cosmetics; (2) quotations of particular interest to us from the Federal Food, Drug, and Cosmetic Act, as amended; and from the Food Additives Amendment of 1958; (3) identifying the Federal agencies—and a few of their key men—responsible for administering these laws; (4) naming a few recent releases of the Food and Drug Administration; (5) mentioning several important "nonprivate" meetings where these various concerned problems were discussed simultaneously—at the same given meeting—by officials of FDA; by experts from various concerned industries; and—in several cases—by "qualified experts in the field" from private research organizations and/or well-known centers of higher professional learning; (6) mentioning certain important books, pamphlets, periodicals, specific technical literature references, and other information sources that have come to my attention, accidentally or otherwise; (7) to mention a very few industrial trade associations, etc., that are—and have been—working diligently on the concerned difficulties insofar as food packaging is involved; (8) a little about what we, personally, as technical men in the paper industry should do in these matters; and (9) a very little about the "prognosis" for the future.

Apparently, with regard to understanding all about food packaging under the various concerned laws, there are no short-cuts. It seems that in order to obtain an adequate knowledge of the subject, one has to do a lot of reading and listening—and discarding of chaff—and then finally ask the right people the right questions at the right time. What has surprised me is that after one really gets into this subject, things become extremely interesting. Then, also, the many apparent inconsistencies become clarified to where one can see the woods in spite of the trees. One personal opinion I shall express without hesitation: I am fully convinced that the rank and file of the American paper industry is—and long has been—most anxious to do everything in its power to promote public health and to conform with all of the concerned laws and regulations, both State and Federal. The significant part that paper packaging has played in reducing illness due to spoiled and/or biologically infected food is well-known to all public health authorities. Apparently, no instances have been reported where food has been poisoned by its paper package, or paper wrapper.

HISTORY

During the period surrounding the Spanish-American War, and particularly between 1898 and 1906, the prolonged efforts of Dr. Harvey W. Wiley (born 1844, died 1930), for many years chief chemist of the U. S. Department of Agriculture—and still regarded as one of the world's greatest workers in the fields of food standards, food protection, etc.—culminated in the enactment of the Federal Pure Food and Drug Act of 1906 (signed by President Theodore Roosevelt on June 30, 1906), and in the Meat Inspection Act (also signed on June 30, 1906). Both of these laws became effective in 1907.

Whereas, several additional legislative items in the concerned field were enacted subsequently, there was no general revision of the Pure Food and Drug Act of 1906 until the enactment of the Federal Food, Drug, and Cosmetic Act of 1938.

Several factors contributed toward the enactment of

Anthony W. Pesch, International Paper Co., Southern Kraft Div., Mobile, Ala.

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Only a plant trial, however, can prove how Westvaco Soda Ash can simplify and speed up your processing and better your end-products.

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the 1938 Act, particularly some controversies regarding certain types of new products that were appearing at, apparently, accelerated rates. There was apprehension, in some quarters, that some new items were not being checked adequately enough to insure public safety. Some of the details of that history were contained in the paper on Food Additives Amendment matters that John A. Quill, FDA, presented before the Chicago Section of TAPPI on the night of Dec. 1, 1959. Mr. Quill, I believe, is head of the Chicago Field Office of the U. S. Food and Drug Administration. At that time I asked Mr. Quill if I might use a copy of his paper in connection with my own proposed technical paper for tonight. Mr. Quill did not have an available copy; but on Dec. 5, 1959, I received, from Mr. Quill's office in Washington, copies of the two recent papers by two FDA officials that Mr. Quill had used as source material for his talk before the Chicago Section of TAPPI on December 1. I shall identify those two papers later (one by Arthur A. Checchi, FDA, the other by John L. Harvey, FDA).

In any event, with regard to the over-all matters involved with this present paper, the following Federal laws (and proposed laws), apparently, are of significant importance (I have not read all of them):

1. The Federal Food, Drug, and Cosmetic Act, as Amended;
2. The Meat Inspection Act;
3. The Poultry Products Inspection Act;
4. The Pesticides Amendment (the "Miller Act ");
5. The Food Additives Amendment of 1958;
6. The Amendment (Regarding Oranges; Food Coloring) Approved March 17, 1959; this act is also officially identified as Public Law 86-2, 86th Congress, S. 79, March 17, 1959;
7. Several items of proposed legislation now before Congress, one of them reportedly being spoken of as the Color Additives Amendment.

In the remainder of this paper, insofar as federal laws are concerned, I shall confine myself almost entirely with items (1) and (5). But I suggest that those of you, whose duties are here involved, also inform yourselves (and keep yourselves informed) with regard to (2), (3), (4), (6), and (7).

Insofar as papers and paperboards are concerned, the reported present opinion of FDA seems to be that any paper that comes in contact with food at any time is food packaging paper. Food packaging paper may or may not contain a food additive. Only those food packaging papers which contain a food additive come within the scope of the Food Additives Amendment. If such paper can be shown to have been manufactured with the intent that it would be used for food packaging purposes, and it can be demonstrated to contain unsafe substances which do contaminate the food and make it unsafe for consumption, it is subject to seizure under the Federal Food, Drug, and Cosmetic Act as soon as the paper manufacturer or converter introduces it into commerce (interstate or intrastate) for ultimate use in packaging foods which are known to be introduced into interstate commerce.

FEDERAL FOOD, DRUG, AND COSMETIC ACT, AS AMENDED

The specific "version" from which I shall quote briefly is further identified on the front cover page by the following: "U. S. Department of Health, Education, and Welfare—Food and Drug Administration—F. D. C. Act, June 1958 Revision." The following appears on the inside of the front cover page: "For sale by the Superintendent of Documents, U. S. Government Printing Office, Washington 25, D. C.—Price 35 cents." Under "Contents" on that same page the following items (among others) appear and seem, in part, particularly significant to us as technical men in the paper industry.

"Definitions" ..... Section 201;
"Prohibited acts and penalties" (under seven categories, Sections 301–307);
"Food" (under eight categories, Sections 401–408);
"General administrative provisions" (under eight categories, Sections 701–707); and
"Miscellaneous" (under two categories, Sections 901 and 902).

In line with what I have already said, the following quotations from the above version of the Act seem to be of paramount significance (to us as technical men in pulp and paper).

"Chapter II—Definitions":

"Sec. 201. For the purposes of this Act—

"(f) The term 'food' means (1) articles used for food or drink for man or other animals, (2) chewing gum, and (3) articles used for components of any such article."

"Sec. 201 (q). The term 'pesticide chemical' means any substance which, alone, in chemical combination or in formulation with one or more other substances, is an 'economic poison' within the meaning of the Federal Insecticide, Fungicide, and Rodenticide Act (7 U.S.C. secs. 135–135K) as now in force or as hereafter amended, and which is used in the production, storage, or transportation of raw agricultural commodities."

"Sec. 201 (r). The term 'raw agricultural commodity' means any food in its raw or natural state, including all fruits that are washed, colored, or otherwise treated in their unpeeled natural form prior to marketing."

Immediately after Section 201 (r) the following is included among other bracketed items.

"The following additional definitions for food are provided for in other acts ..... Sec. 201b. Package. The Act of July 24, 1919 (41 Stat. 271), declares "The word "package" where it occurs in this chapter shall include and shall be construed to include wrapped meats inclosed in papers or other materials as prepared by the manufacturers thereof for sale.' "

"Chapter IV—Food":

"Adulterated Food":

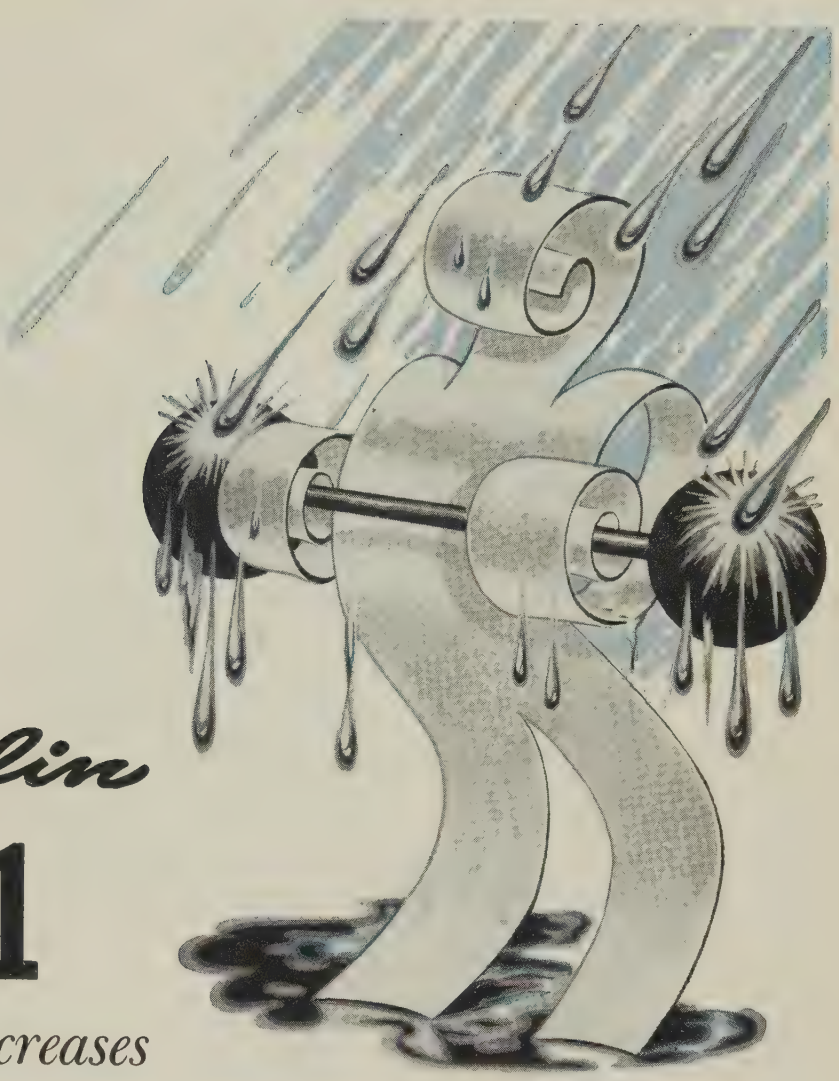
"Sec. 402. A food shall be deemed to be adulterated—

"(a) (1) If it bears or contains any poisonous or deleterious substance which may render it injurious to health; but in case the substance is not an added substance such food shall not be considered adulterated under this clause if the quantity of such substance in such food does not ordinarily render it injurious to health; or

"(2) If it bears or contains any added poisonous or added deleterious substances, except a pesticide chemical in or on a raw agricultural commodity, which is unsafe within the meaning of section 406, or if it is a raw agricultural commodity and it bears or contains a pesticide chemical which is unsafe within the meaning of section 408 (a);

"(3) if it consists in whole or in part of any filthy, putrid, or decomposed substance, or if it is otherwise unfit for food; or

"(4) if it has been prepared, packed, or held under un-



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sanitary conditions whereby it may have become contaminated with filth, or whereby it may have been rendered injurious to health; or

"(5) if it is, in whole or in part, the product of a diseased animal or of an animal which has died otherwise than by slaughter; or

"(6) if its container is composed, in whole or in part, of any poisonous or deleterious substance which may render the contents injurious to health.

"(c) If it bears or contains a coal-tar color other than one from a batch that has been certified in accordance with regulations as provided by section 406: Provided, That this paragraph shall not apply to citrus fruit bearing or containing a coal-tar color if application for listing of such color has been made under this Act and such application has not been acted on by the Secretary, if such color was commonly used prior to the enactment of this Act for the purpose of coloring citrus fruit: Provided further, That this paragraph shall not apply to oranges meeting minimum maturity standards established by or under the laws of the States in which the oranges were grown and not intended for processing (other than oranges designated by the trade as 'packing house elimination'), the skins of which have been colored at any time prior to March 1, 1959, with the coal-tar color certified prior to the enactment of this proviso as F.D.&C. Red 32, or certified after such enactment as External D.&C. Red 14 in accordance with section 21, Code of Federal Regulations, part 9: and Provided further, That the preceding proviso shall have no further effect if prior to March 1, 1959, another coal-tar color suitable for coloring oranges is listed under section 406."

Note 1: Section 402 (a) (3) above, apparently, includes malodors that the food actually absorbed from its surroundings, including any of its packaging (including from outer packaging materials *not in direct contact* with the concerned food).

Note 2: I included Section 402 (c) in the above quotation, even though I do not really understand it (or the significance of the special Amendment (Regarding Oranges; Food Coloring) Approved March 17, 1959, or the significance of the recent reported *de-certifications* of certain coloring materials by FDA). I really hope that somebody will cooperate and help to clarify this whole colors problem for me, particularly the extent to which it applies to food packaging papers of any and all kinds).

"Misbranded Food":

"Sec. 403. A food shall be deemed to be misbranded—

"(d) If its container is so made, formed, or filled as to be misleading.

"(k) If it bears or contains any artificial flavoring, artificial coloring, or chemical preservative, unless it bears labeling stating tht fact: Provided, That to the extent that compliance with the requirements of this paragraph is impracticable, exemptions shall be established by regulations promulgated by the Secretary. The provisions of this paragraph and paragraphs (g) and (i) with respect to artificial coloring shall not apply in the case of butter, cheese, or ice cream."

"Tolerances for Poisonous Ingredients in Food and Certification of Coal-Tar Colors for Food":

"Sec. 406 (a) Any poisonous or deleterious substance added to any food, except where such substance is required in the production thereof or cannot be avoided by good manufacturing practice shall be deemed to be unsafe for purposes of the application of clause (2) of section 402 (a); but when such substance is so required or cannot be so avoided, the Secretary shall promulgate regulations limiting the quantity therein or thereon to such extent as he finds necessary for the protection of public health, and any quantity exceeding the limits so fixed shall also be deemed to be unsafe for purposes of the application of clause (2) of section 402 (a). While such a regulation is in effect limiting the quantity of any such substance in the case of any food, such food shall not, by reason of bearing or containing any added amount of such substance, be considered to be adulterated within the meaning of clause (1) of section 402 (a). In determining the quantity of such added substance to be tolerated in or on different articles of food the Secretary shall take into account the extent to which the use of such substance is required or cannot be avoided in the production of each such article, and the other ways in which the consumer may be affected by the same or other poisonous or deleterious substances.

"(b) The Secretary shall promulgate regulations providing for the listing of coal-tar colors which are harmless and suitable for use in food and for the certification of batches of such colors, with or without harmless diluents."

"Tolerances for Pesticide Chemicals in or on Raw Agricultural Commodities":

Insofar as the involvement of food *packaging* materials under this heading will possibly be relatively small—and because I, myself, am not well enough informed on the various pesticides problems—I shall not quote any portions of the Act under this heading.

"Chapter V—Drugs and Devices":

"Chapter VI—Cosmetics":

Both of these chapters of the Act contain provisions with regard to adulteration from the package. But since the Food Additives Amendment of 1958, apparently, does not apply to Drugs and Cosmetics, I shall not quote any sections of the Act that appear under said two Chapters V and VI.

"Chapter VII—General Administrative Provisions"

"Chapter VIII—Imports and Exports"

"Chapter IX—Miscellaneous"

Note: Probably somebody at every plant making food packaging items should read these last three chapters, particularly somebody in the legal department. I, myself, have not read them.

Stapled to my copy of the above identified version of the Federal Food, Drug, and Cosmetic Act, As Amended is a copy of a separate booklet entitled "General Regulations for the Enforcement of the Federal Food, Drug, and Cosmetic Act—Title 21, Part 1—U. S. Department of Health Education, and Welfare—Food and Drug Administration—F. D. C. Regs., Part 1—June 1958 Revision."

That item is also for sale by the Superintendent of Documents— at 25 cents. Unfortunately, I have not yet studied this item. But I do suggest that we all regard it as "required reading."

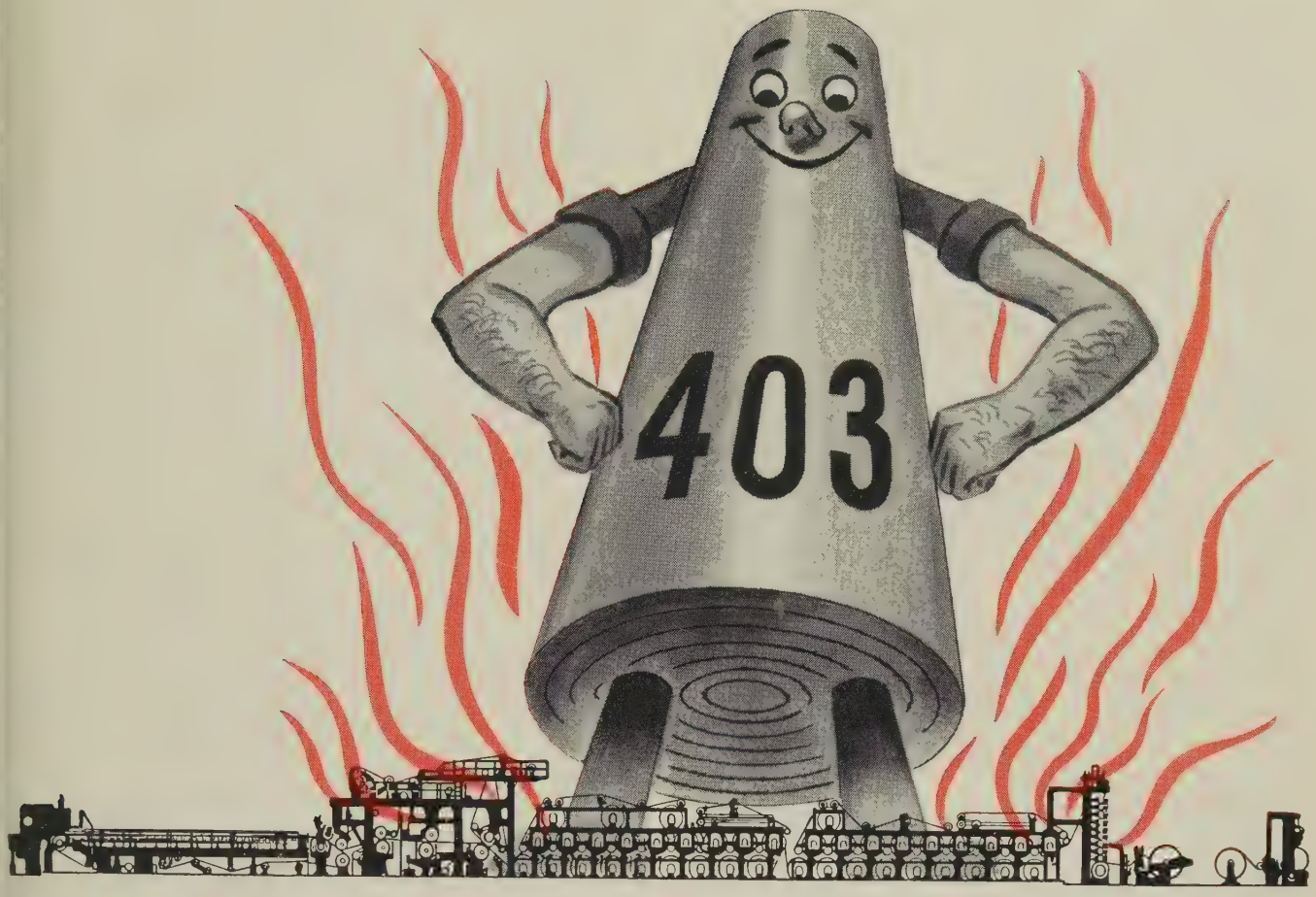
Food Additives Amendment of 1958

This Act (Public Law 85 - 929, 85th Congress, H. R. 13254, Sept. 6, 1958) is of such importance that reference should be made to the official version. For your immediate convenience, however, I am now quoting a few excerpts from the Act—namely, those that it is particularly important for us, as paper industry "scientists," with which to be familiar. But please be informed that italics that appear in these quotations from the Act are my own:

"An Act"

To protect the public health by amending the Federal Food, Drug, and Cosmetic Act to prohibit the use in food of additives which have not been adequately tested to establish their safety."

Be it enacted by the Senate and House of Representatives of the United States of America in Congress assembled



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That this Act may be cited as the "Food Additives Amendment of 1958."

Sec. 2. Section 201, as amended, of the Federal Food, Drug, and Cosmetic Act is further amended by adding at the end of such section the following new paragraphs:

"(s) The term 'food additive' means any substance the intended use of which results or may reasonably be expected to result, directly or indirectly, in its becoming a component or otherwise affecting the characteristics of any food (including any substance intended for use in producing, manufacturing, packing, processing, preparing, treating, packaging, transporting, or holding food; and including any source of radiation intended for any such use), if such substance is not generally recognized, among experts qualified by scientific training and experience to evaluate its safety, as having been adequately shown through scientific procedures (or, in the case of a substance used in food prior to Jan. 1, 1958, through either scientific procedures or experience based on common use in food) to be safe under the conditions of its intended use; except that such term does not include—

"(1) a pesticide chemical in or on a raw agricultural commodity; or

"(2) a pesticide chemical to the extent that it is intended for use or is used in the production,s torage, or transportation of any raw agricultural commodity; or

"(3) any substance used in accordance with a sanction or approval granted prior to the enactment of this paragraph pursuant to this Act, the Poultry Products Inspection Act (21 U.S.C. 451 and the following) or the Meat Inspection Act of March 4, 1907 (34 Stat. 1260), as amended and extended (21 U.S.C. 71 and the following).

"(t) The term 'safe,' as used in paragraph (s) of this section and in section 409, has reference to the health of man or animal."

Sec. 3. (a) Clause (2) of section 402 (a), as amended, of such Act is amended to read as follows: "(2) (A) if it bears or contains any added poisonous or added deleterious substance (except a pesticide chemical in or on a raw agricultural commodity and except a good additive) which is unsafe within the meaning of section 406, or (B) if it is a raw agricultural commodity and it bears or contains a pesticide chemical which is unsafe within the meaning of section 408 (a), or (C) if it is, or it bears or contains, any food additive which is unsafe within the meaning of section 409: Provided, That where a pesticide chemical has been used in or on a raw agricultural commodity in conformity with an exemption granted or a tolerance prescribed under section 408 and such raw agricultural commodity has been subjected to processing such as canning, cooking, freezing, dehydrating, or milling, the residue of such pesticide chemical remaining in or on such processed food shall, notwithstanding the provisions of sections 406 and 409, not be deemed unsafe if such residue in or on the raw agricultural commodity has been removed to the extent possible in good manufacturing practice and the concentration of such residue in the processed food when ready to eat is not greater than the tolerance prescribed for the raw agricultural commodity;."

(b) Section 402 (a), as amended, of such Act is further amended by striking out the period at the end thereof and inserting in lieu thereof a semicolon and the following: "or (7) if it has been intentionally subjected to radiation, unless the use of the radiation was in conformity with a regulation or exemption in effect pursuant to section 409."

(c) The first sentence of section 406 (a) of such Act is amended by striking out "clause (2)" wherever it appears in such sentence and inserting in lieu thereof "clause (2) (A)."

Sec. 4. Chapter IV of such Act is amended by adding at the end thereof the following new section:

"FOOD ADDITIVES"

"Unsafe Food Additives"

"Sec. 409. (a) A food additive shall, with respect to any particular use or intended use of such additives,be deemed to be unsafe for the purposes of the application of clause (2) (C) of section 402 (a), unless—

"(1) it and its use of intended use conform to the terms of an exemption which is in effect pursuant to subsection (i) of this section; or

"(2) there is in effect, and it and its use or intended use are in conformity with, a regulation issued under this section prescribing the conditions under which such additive may be safely used.

While such a regulation relating to a food additive is in effect, a food shall not, by reason of bearing or containing such an additive in accordance with the regulation, be considered adulterated within the meaning of clause (1) of section 402 (a).

"Petition to Establish Safety

"(b) (1) Any person may, with respect to any intended use of a food additive, file with the Secretary a petition proposing the issuance of a regulation prescribing the conditions under which such additive may be safely used.

"Action on the Petition"

"(c) (1) The Secretary shall—

"(3) No such regulation shall issue if a fair evaluation of the data before the Secretary—

"(A) fails to establish that the proposed use of the food additive, under the conditions of use to be specified in the regulation, will be safe: *Provided, That no additive shall be deemed to be safe if it is found to induce cancer when ingested by man or animal, or if it is found, after tests which are appropriate for the evaluation of the safety of food additives, to include cancer in man or animal;* or

"(B) shows that the proposed use of the additive would promote deception of the consumer in violation of this Act or would otherwise result in adulteration or in misbranding of food within the meaning of this Act.

"(4) If, in the judgment of the Secretary, based upon a fair evaluation of the data before him, a tolerance limitation is required in order to assure that the proposed use of an additive will be safe, the Secretary—

"(A) shall not fix such tolerance limitation at a level higher than he finds to be reasonably required to accomplish the physical or other technical effect for which such additive is intended; and

"(B) shall not establish a regulation for such proposed use if he finds upon a fair evaluation of the data before him that such data do not establish that such use would accomplish the intended physical or other technical effect.

"(5) In determining, for the purposes of this section, whether a proposed use of a food additive is safe, the Secretary shall consider among other relevant factors—

"(A) the probable consumption of the additive and of any substance formed in or on food because of the use of the additive;

"(B) *the cumulative effect of such additive in the diet of man or animals, taking into account any chemically or pharmacologically related substance or substances in such diet;* and

"(C) safety factors which in the opinion of experts qualified by scientific training and experience to evaluate the safety of food additives are generally recognized as appropriate for the use of animal experimentation data.

Sec. 6. (a) Except as provided in subsections (b) and (c) of this section, this Act shall take effect on the date of its enactment.

(b) Except as provided in subsection (c) of this section, section 3 of this Act shall take effect on the one hundred and eightieth day after the date of enactment of this Act.

(c) With respect to any particular commercial use of a

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food additive, if such use was made of such additive before Jan. 1, 1958, section 3 of this Act shall take effect—

(1) either (A) one year after the effective data established in subsection (b) of this section, or (B) at the end of such additional period (but not later than two years from such effective date established in subsection (b) as the Secretary of Health, Education, and Welfare may prescribe on the basis of a finding that such extension involves no undue risk to the public health and that conditions exist which necessitate the prescribing of such an additional period, or

(2) on the date on which an order with respect to such use under section 409 of the Federal Food, Drug, and Cosmetic Act becomes effective, whichever date first occurs.

Sec. 7. Nothing in this Act shall be construed to exempt any meat or meat food product or any person from any requirement imposed by or pursuant to the Poultry Products Inspection Act (21 U.S.C. 451 and the following) or the Meat Inspection Act of March 4, 1907, 34 Stat. 1260, as amended and extended (21 U.S.C. 71 and the following)."

Note 1: The first italicized section in the above quotations from the Food Additives Amendment of 1958 represents what has become known as the *"Delaney Clause"* (named after Congressman James J. Delaney—Representative from New York, Seventh District—Queens County—whose biography appears on Page 102, "Congressional Directory," 86th Congress, 1st Session, March 1959—and whose name is also listed on page 252 of the same issue of the "Congressional Directory" as a member of the House Rules Committee. Reportedly, the current FDA interpretation of the so-called Delaney Clause is that the tolerance for any such substances added to food is zero (meaning absolutely zero). This, reportedly, is causing very serious chemical analysis problems, particularly because, while the most sensitive presently available analytical procedure for the alleged suspect might "show" it absent today, a possible future more sensitive analytical method for the same alleged suspect might show it to be present in the same identical sample next year—or, say, ten years from now. Apparently, similar analytical difficulties *may also arise* with regard to certain other substances, such as those concerned in the second portion that I have italicized, or for possible other *added substances* for which a *zero* tolerance may be imposed, now or in the future.

Note 2: In connection with Note 1 above, it is suggested that you examine the *"revised"* so-called "FDA white list" that was published in the Nov. 20, 1959, issue of the "Federal Register," particularly with regard to six identified items formerly "approved" but now deleted ("approval" withdrawn). Said deletions were, apparently, made because *certain grades only* of those six commodities may, possibly, contain objectionable impurities, even though the chief component in each case may, apparently, be regarded as safe. Reportedly, in the opinion of experts, it has been suggested that, in such cases, it is advisable to consult with the specific suppliers concerned and the specifically concerned officials of the FDA.

FEDERAL ADMINISTRATIVE AGENCIES

The principal officers, officials, and agencies concerned with the administration of the laws referred to above are, I believe, the following:

a. *Department of Health, Education and Welfare.—Arthur S. Fleming Secretary*
- A. *Food and Drug Administration**
  - Commissioner—George P. Larrick
  - Deputy Commissioner—John L. Harvey
  - Scientific Director—Paul L. Day
  - Executive Officer—Leo L. Miller
  - Director of—
    - Division of Federal-State Relations—James C. Pearson
    - Division of Public Information—Wallace F. Janssen
    - Bureau of Biological and Physical Sciences—Robert S. Roe
      - Division of Antibiotics—Henry Welch
      - Division of Cosmetics—G. Robert Clark
      - Division of Food—O. L. Kline
      - Division of Microbiology—Glenn G. Slocum
      - Division of Nutrition—(Vacant)
      - Division of Pharmaceutical Chemistry—Frank H. Wiley
      - Division of Pharmacology—Arnold J. Lehman
    - Bureau of Enforcement—Malcolm R. Stephens
      - Division of Regulatory Management—Kenneth L. Milstead
    - Bureau of Field Administration—Allan E. Rayfield
    - Bureau of Medicine—William H. Kessenich
    - Bureau of Program Planning and Appraisal—Shelbey T. Grey

b. *Department of Agriculture—Ezra Taft Benson, Secretary*

Note: I am not now sufficiently informed to say anything more regarding to what extent the U. S. Department of Agriculture exercises regulatory powers with regard to food packaging materials, excepting that one of its regulatory divisions, the Meat Inspection Division (A. R. Miller, Director), is probably involved.

RECENT RELEASES, ETC. BY THE FOOD AND DRUG ADMINISTRATION

In view of the complexities, etc., involved in the administration of the Food Additives Amendment it is, perhaps, both unfair and hazardous for men in our industry to base their conclusions on unofficial sources or on some of the early reported views of the Food and Drug Administration. I suggest that you obtain copies of the following three very recent releases (a, b, and c) from the Department of Health, Education, and Welfare, Washington 25, D. C.; also that you obtain copies (from some source) of the "safe" items listed in the releases identified in (d), (e), and (f) below.

a. *Food Packaging Under the Food Additives Amendment—What Needs to Be Done,"* by Arthur A. Checchi, Assistant to the Deputy Commissioner, Food and Drug Administration ("For presentation at the 14th Annual Paper and Plastics Conference, Edgewater Beach Hotel, Chicago, Ill., Sept. 22, 1959").

b. *"Experience with the Food Additives Amendment,"* John L. Harvey, Deputy Commissioner, Food and Drug Administration ("For Release Upon Delivery"—"For delivery at the American Public Health Association Convention in Atlantic City, N. J., Oct. 20, 1959").

Note: Some of you probably heard Mr. Checchi's paper at the concerned TAPPI Fourteenth Paper-Plastics Conference. Mr. Checchi retired to private business recently. I had intended, originally, to quote excerpts from both Mr. Harvey's paper and Mr. Checchi's paper, but limitations of space and time have made that impracticable. In any event, I feel that both papers, *complete,* are "required reading" on these matters.

c. *"Incidental Food Additives,"* by Winton B. Rankin, Assistant to the Commissioner, Food and Drug Administration ("For delivery at Food and Drug Administration—Food Law Institute Conference, Washington, D. C., Nov. 16 and 17, 1959").

Note: I regard the reading of the paper by Mr. Rankin as another "must" item.

d. *The Specific List of "Substances that were proposed for classification as generally recognized as safe"* that was published in the "Federal Register" of Dec. 9, 1958.

e. *The Specific List of "Substances that are proposed for classification as generally recognized as safe"*—"Spices and Other Natural Seasonings (Leaves, Roots, Barks, Berries, etc.)" . . . . . that was published in the "Federal Register" of April 21, 1959.

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* Names and titles taken from "Congressional Directory," 86th Congress, 1st Session, March, 1959. But Dr. M. Fischbach is now Chief, Division of Food and Dr. O. L. Kline is now Chief, Division of Nutrition.

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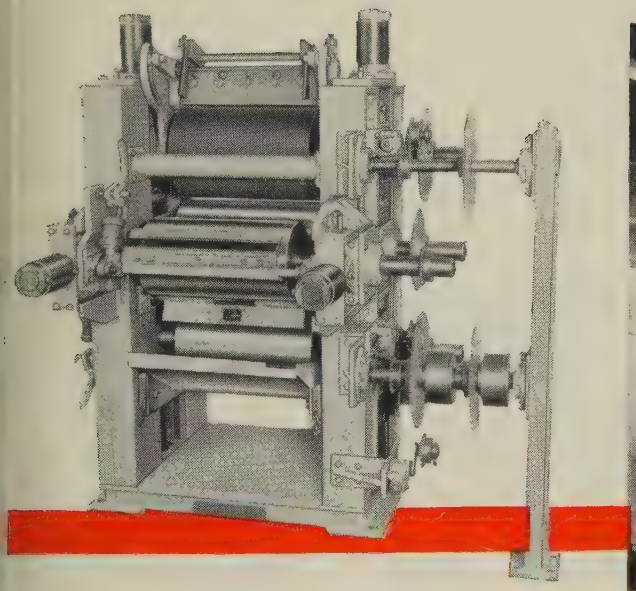

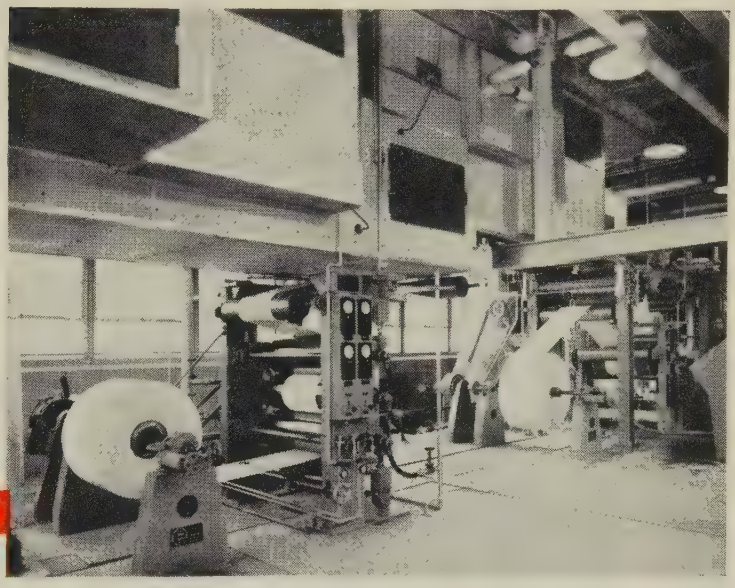

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f. *The Specific List of "Substances that are generally recognized as safe"* that was published in the "Federal Register" of Nov. 20, 1959.

Note: As already explained earlier in this paper, it "withdraws the approval" of six specific substances that had previously been "approved" per item (d) above.

SOME IMPORTANT TECHNICAL MEETINGS WHERE FDA OFFICIALS WERE ALSO PRESENT

a. *Meeting of the Packaging Institute,* Hotel Statler, New York, N. Y., March 3, 1959. Several of the technical papers, etc., only presented at the New York, and subsequent meetings were published Dec. 10, 1959 by the Packaging Institute.

b. *Special Course in Food Technology (20.82S Food Technology), Massachusetts Institute of Technology,* Cambridge, Mass., June 22–26, 1959. To me, personally, this was the most valuable, interesting, informative 5-day course of instruction that I, myself, have ever had the privilege to attend (5 full days of concentrated effort on the part of both "faculty" and "students"). About 58 students were enrolled, many of them prominent persons from industry, educational institutions, the military, governmental career men, etc.—plus a few technical men from the paper industries. About 27 prominent men from many fields connected with foods, food processing, food regulations, etc., spoke upon many food technology problems, including food packaging and the Food Additives Amendment. A number of these speakers were key men with FDA and other governmental agencies, Federal, State, and Foreign. During that same week the Association of Food and Drug Officials of the United States had its annual convention in Boston, Mass. A number of us attended the annual banquet of said Association (which includes both State and Federal public health officials). At that banquet Arnold J. Lehman, M.D., Chief, Division of Pharmacology, Food and Drug Administration, was signally honored for some of his outstanding specific contributions in the public health field.

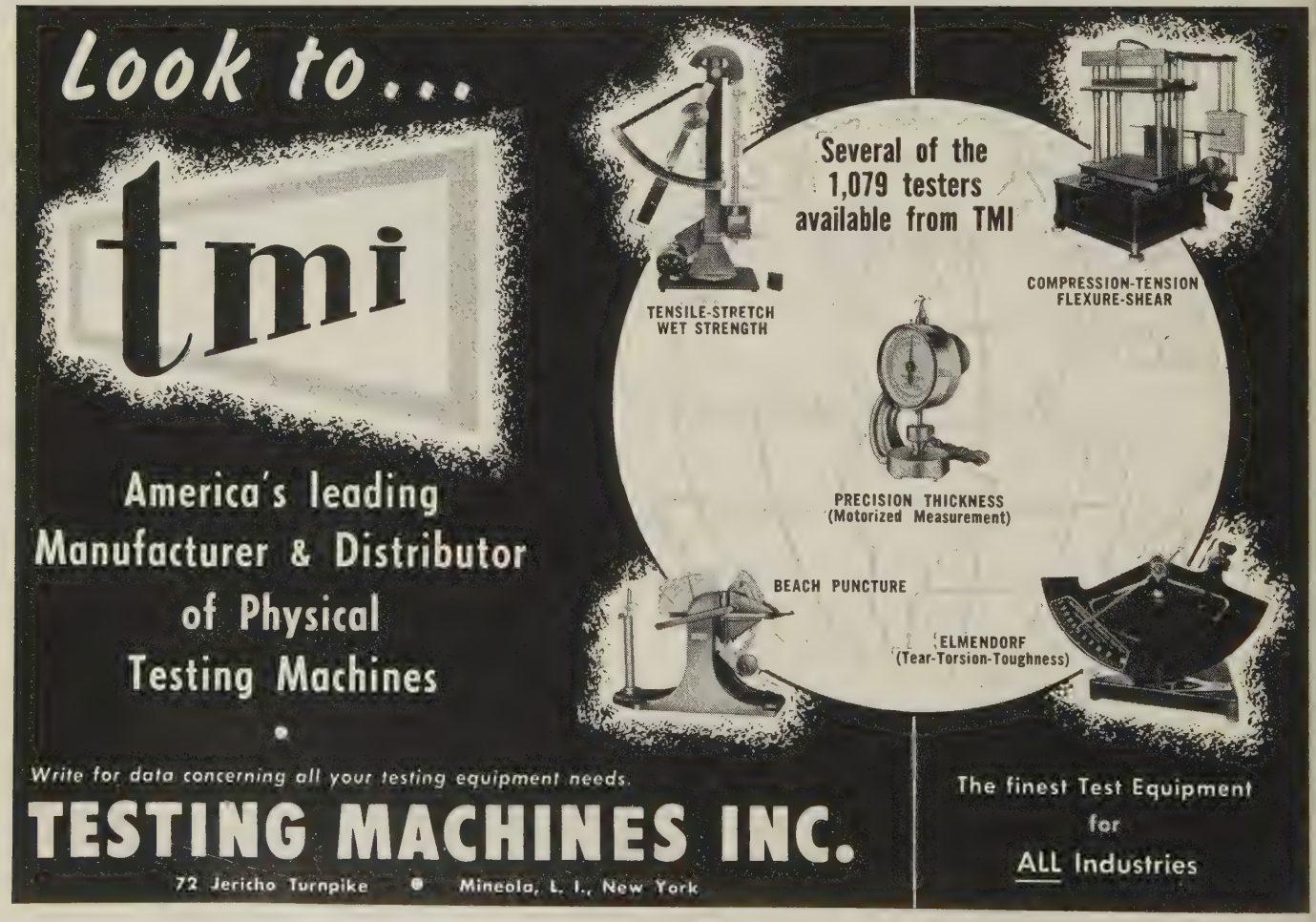
This special Summer Session course was conducted by the late Dr Bernard E. Proctor, Head, Department of Food Technology, M.I.T., and Dr. S. A. Goldblith, Executive Officer, Food Technology, M.I.T. Professor Proctor was a current member of the Food Technology Subcommittee, Food Protection Committee, National Research Council.

I have no knowledge regarding whether or not any similar course will be given by Massachusetts Institute of Technology in the future.

c. *Eleventh Annual Conference of the Association of Food and Drug Officials of the Southern States,* Battle House Hotel, Mobile, Ala., April 27–29, 1959. Some of you that are present tonight may also have been at that meeting. One of the speakers was James C. Pearson, Director, Division of Federal-State Relations, Food and Drug Administration.

d. *TAPPI Fourteenth Paper-Plastics Conference,* Edgewater Beach Hotel, Chicago, Ill., Sept. 21–23, 1959. This meeting was referred to above. I did not attend that meeting.

e. *Meeting of the Packaging Institute (Packaging Institute's 21st Annual National Packaging Forum),* Hotel Statler, New York, N. Y., Nov. 16–18, 1959. One of the speakers at that meeting was John L. Harvey, Deputy Commissioner, Food and Drug Administration. I did not attend that meeting.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Obviously, any appearance of slime in a mill system calls for *positive identification* of its substance, and *action* to bring it under control.

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Nalco Field Service Men have the training and experience needed to deal quickly and effectively with paper mill slimes. They carry test equipment to analyze your system . . . not only for microbiological activity, but also for the many organic and inorganic elements that are part of most deposits. Supplementing their on-the-spot service is the Nalco Paper Process Laboratory, a service and research center equipped for the most exacting analysis work and for comprehensive research on every phase of pulp and paper mill slime control.

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f. *Second Annual Food and Drug Administration—Food Law Institute (FLI) Conference,* held in the Auditorium of the Department of Health, Education, and Welfare, Washington, D. C., Nov. 16 and 17, 1959. Regarding the papers and discussions at this very important meeting I suggest very strongly that you consult such periodicals as the Nov. 23, 1959, issue of the weekly newsletter, *Food Chemical News,* and the Nov. 30, 1959, issue of the magazine, *Chemical and Engineering News.* I did not attend that meeting.

g. *Meeting of the Chicago Section of TAPPI,* Chicago Bar Association Building, Chicago, Ill., Dec. 1, 1959. In "History" I have already spoken about the talk that was made at that meeting by John A. Quill, U. S. Food and Drug Administration, Chicago Field Office.

REFERENCES AND REFERENCE SOURCES

The following books, technical papers, periodicals, sources, etc., are items of special interest that have come to my attention more or less accidentally. Certainly there must be many other very important items and sources of information in these matters about which I now have little, or no, knowledge. "Gold is where you find it."

a. Some standard textbook on food matters, such as the three-volume, 2580- page book, "The Chemistry and Technology of Food and Food Products," Morris B. Jacobs, Ph.D., Editor, Second Edition (1951), Interscience Publishers, Inc., New York, N. Y.

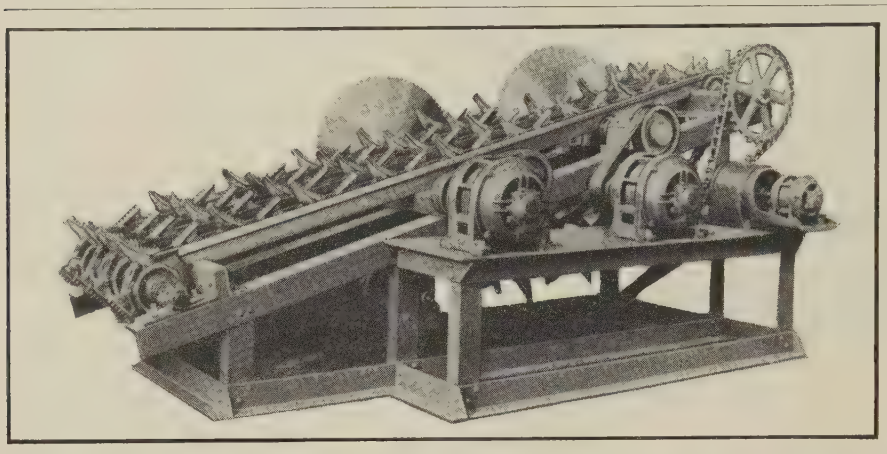
b. The paper-bound book (approximately 106 pages), "Appraisal of the Safety of Chemicals in Food, Drugs, and Cosmetics," Edited and Published by The Editorial Committee, Association of Food and Drug Officials of the United States, Editorial Office, 2411 N. Charles St., Baltimore 18, Md. This is an "absolute must" item.

c. The paper-bound book (approximately 408 pages), "Handbook of Toxicology, Volume I, Acute Toxicities," Edited by William S. Spector, (1956), W. B. Saunders Co., Philadelphia, Pa.

d. The 48-page booklet, "Food Packaging Materials, Their Composition and Uses"—A Report of the Food Protection Committee, Food and Nutrition Board" (of the National Academy of Science-National Research Council). This is Publication 645, National Academy of Sciences-National Research Council, Washington 25, D. C. (November, 1958).

This is another "*absolute must*" item. This publication lists the membership of the Food Protection Committee, and of its Food Technology Subcommittee. As concerned technical men, we need to be familiar with the membership, duties, and advisory capacities of these two committees. This particular publication does not list the names of the men, from various fields, who serve as liaison men to the Food Protection Committee. At this time, I do not happen to know how one may obtain the names, etc., of the men who serve as liaison men to the Food Protection Committee.

e. The weekly news magazine of the American Chemical Society, *Chemical and Engineering News.* In particular, I call your attention to the Nov. 30, 1959, issue, page



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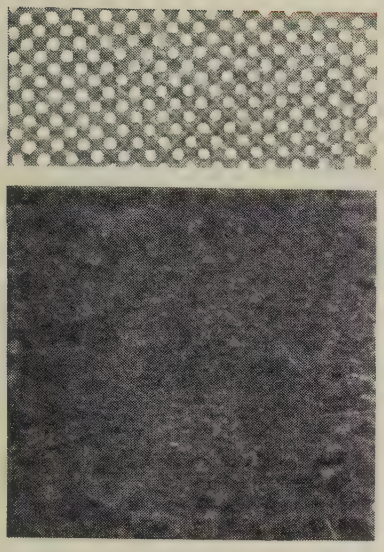

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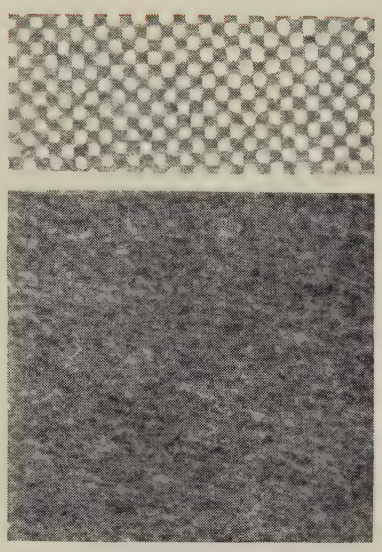

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40, "Government, Industry Airs Additives Problems—Industry may have trouble meeting deadline on additive approvals, FDA-FLI conference hears."

f. The weekly newsletter type periodical, *Food Chemical News*—the publication of which started with the March 16, 1959, issue—particularly, the issue of Nov. 23, 1959 (which also covers the recent FDA-FLI conference) and other recent issues. I, personally, have found this publication *extremely* helpful (Price $150 a year, Additional subscriptions $100).

g. The weekly magazine, *Chemical Week*, published by McGraw-Hill Publishing Co., Inc., 330 W. 42nd St., New York 36, N. Y.

h. The bi-weekly loose leaf service of Commerce Clearing House, Inc., entitled *Food Drug Cosmetic Law Reports.* A principal office of the publisher is at 420 Lexington Ave., New York 17, N. Y. I have not seen any issue of this publication, but very recently it was recommended to me very highly by a friend who, personally, qualifies as one of the "experts qualified by scientific training and experience to evaluate safety" in connection with paper food packaging materials. The price is $105 per year for 2 years, or $120 for 1 year.

i. The *Quarterly Bulletin—Association of Food & Drug Officials of the United States.* Editorial Office, Dr. Wm. F. Reindoller, 301 W. Preston St., Baltimore 1, Md.—Business Office, Joe F. Lakey, Texas State Department of Health, Austin 1, Texas.

Note: This is a "must" item.

j. Probably some of the other periodicals and or publications of the American Chemical Society (besides item (e) above) frequently also carry new analytical procedures and other timely information with regard to food additives problems.

Note: The items through (j) above are not intended to be anything like an all-inclusive references list in these matters. There are many other helpful domestic and foreign books and publications in this field, such as those of the Institute of Paper Chemistry, the American Association for the Advancement of Science, the Society for Chemical Industry (British) etc. I simply have not had sufficient time to locate many of them.

TRADE ASSOCIATIONS ETC., ACTIVELY AT WORK IN CONNECTION WITH CONCERNED PROGRAMS

Reportedly, the officials of the Food and Drug Association often prefer to discuss broad and specific industry programs on matters of this kind with the trade associations of the industries concerned. The following are a very few of the many trade associations that are particularly active in connection with the paper industry programs in food packaging:

a. *Trade Associations of the Paper Industry, Proper:* Reportedly, such agencies as the American Paper and Pulp Association, APPA; the National Paperboard Association, NPA; the Chemical Additives Committee of APPA; the Biological and Chemical Research Committee of NPA; the APPA-NPA Food Additives Liaison Committee; the Boxboard Research and Development Association; and several other paper industry trade associations, some of which are, also, already known to you.

b. *Trade Associations, etc., Among Suppliers to the Paper Industry:* Reportedly, such agencies as the American Petroleum Institute, API;

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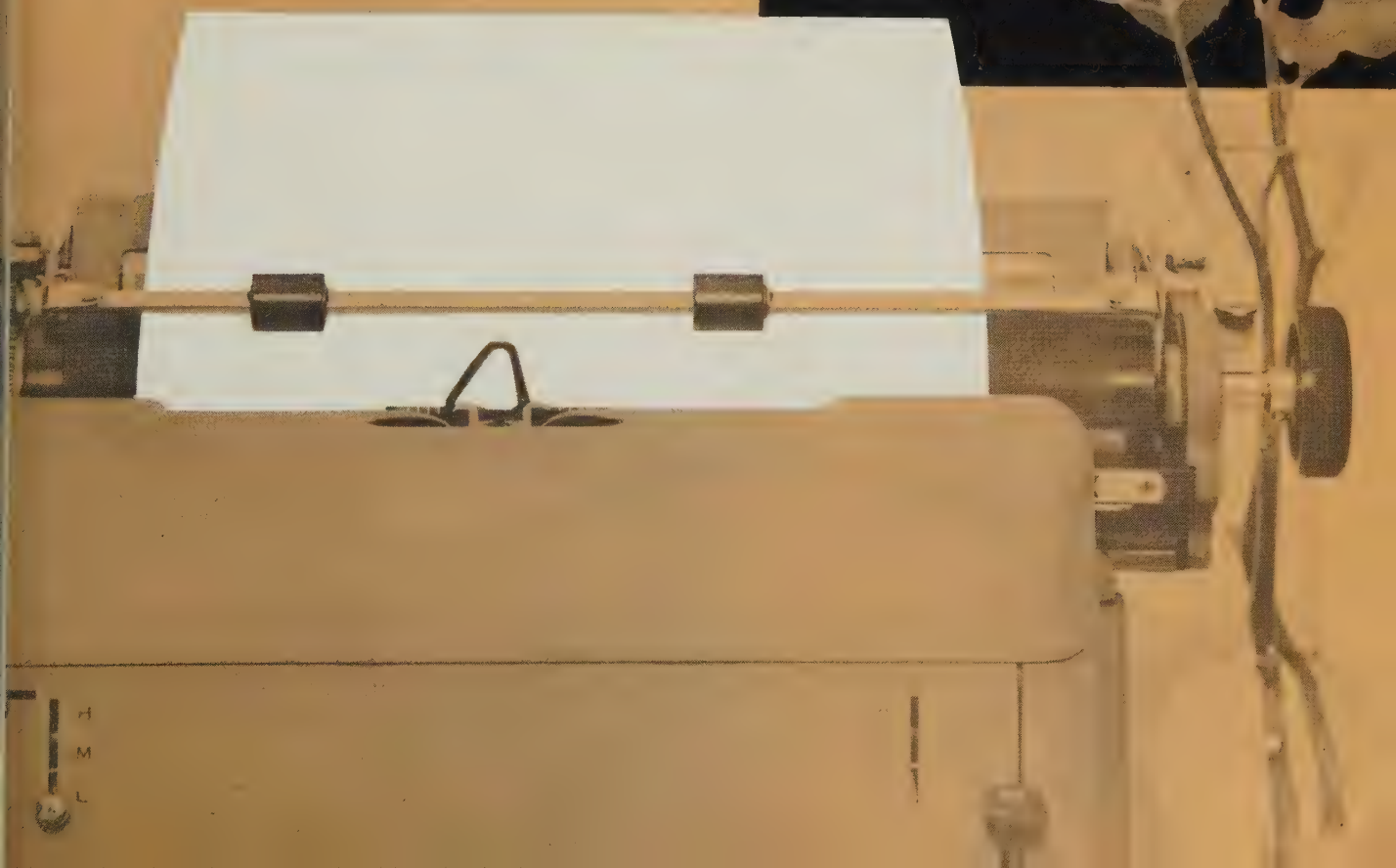

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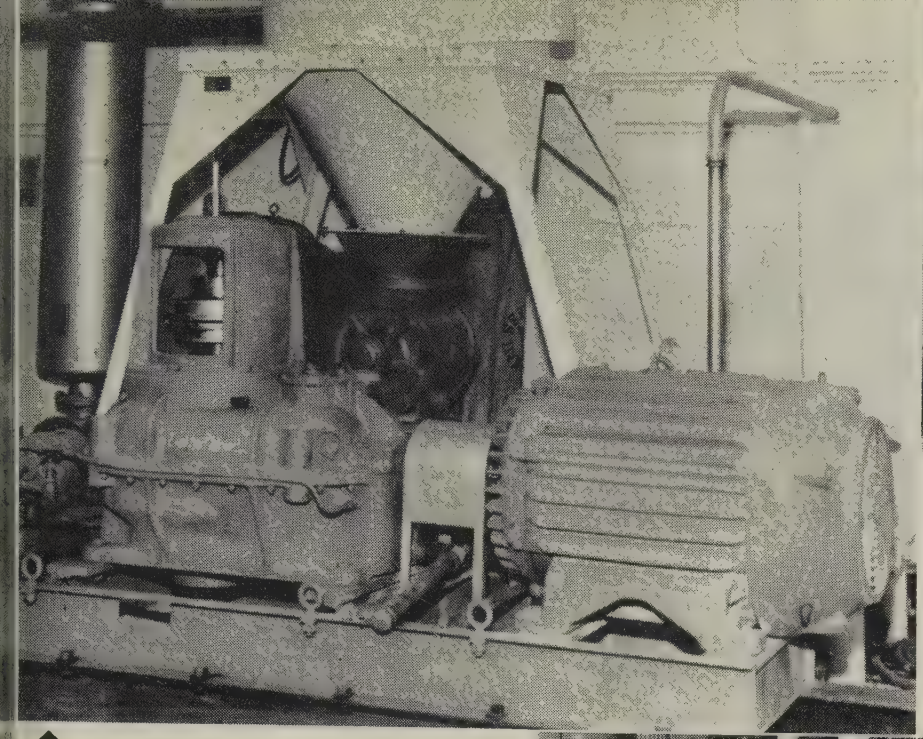

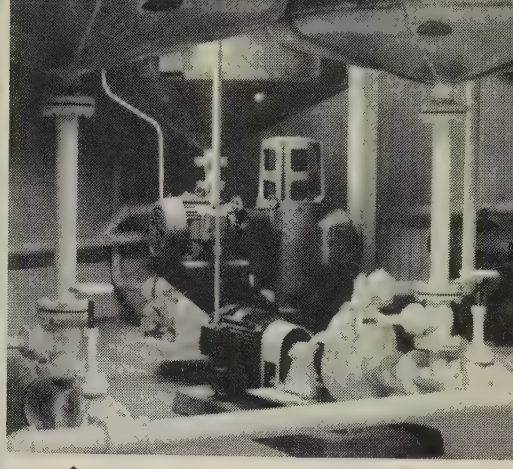

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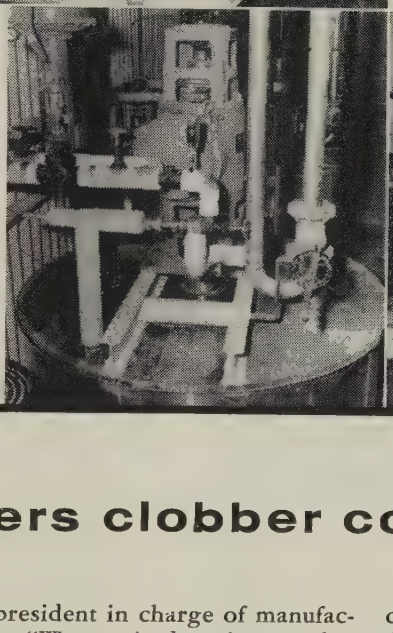
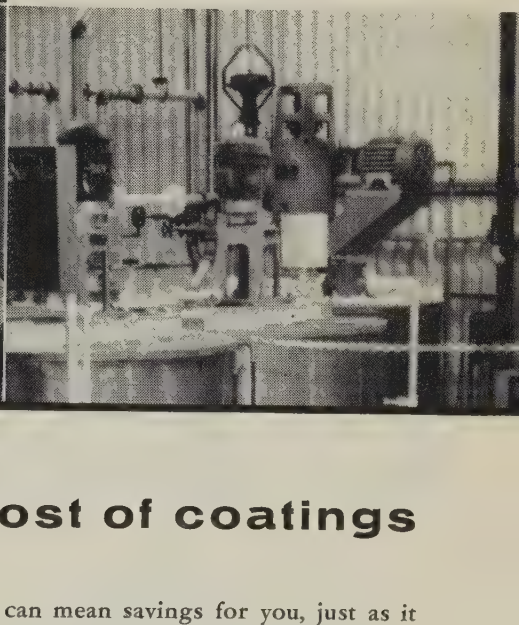
HIGHLY THIXOTROPIC clay-starch slurry from cooker turns over in two tanks equipped with 10-hp LIGHTNIN Mixers. Then it moves to 1,000-gallon storage tanks where 5-hp LIGHTNIN Mixers keep it thoroughly blended to prevent crusting before use.





One man, 20 mixers clobber cost of coatings

Designed to handle clay in bulk as a slurry, this new mixing system at Blandin Paper Company, Grand Rapids, Minn., helps in four ways to prepare coatings at record low cost.

1. The system itself cost much less to build than comparable coating facilities.

2. It permits the purchase of lower-cost bulk clay.

3. Ended are the problems of space-wasting dry storage, handling of bags, and dust. Clay is always ready for immediate use.

4. One man per shift handles all operations—with the help of 20 LIGHTNIN Mixers ranging in size from 3 to 75 hp.

What mill management says

"We knew that mixing was going to be a vital part of our new process," says Harold F. Zigmund, Blandin's vice-president in charge of manufacturing. "We required equipment that was sound and could give us the process result we were looking for.

"LIGHTNIN Mixers have enabled us to accomplish both of our goals. The cooperation and service we have obtained from the MIXCO field representative have been most helpful to us."

Since completing the new coatings plant, Blandin has decided to use LIGHTNIN Paper Stock Agitators on the mill's three brown-stock chests, a neutralization chest, and a broke chest.

Guaranteed mixing performance can mean savings for you, just as it does for Blandin. And it's easy to get. At a call from you, an experienced LIGHTNIN Mixer representative can size up your needs and help you select precisely the right mixing system to deliver the results you want.

He draws upon the experience of thousands of test runs, and successful installations in several hundred mills. He offers you widest possible choice of *standard* power-speed combinations and mixer designs. Call him in now (he's listed in Paper & Pulp Mill Catalog). Or write us direct.

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certain manufacturers of rosin, and rosin sizes; certain manufacturers of wax sizes; the Synthetic Organic Chemical Manufacturers Association, SOCMA; the Corn Industries Research Foundation; and others.

c. *Research Organizations:* The Oct. 31, 1959, issue of the magazine, *Chemical Week*, lists a few of the private and/or institutional research corporations, or research organizations, listed below (the concerned article, "Rush Season to Toxicology Researchers," begins on page 33 of said issue of *Chemical Week*). Neither that listing, apparently, nor the following longer list, pretends to be complete.

A. Mellon Institute, Pittsburg, Pa.
B. Food and Drug Research Laboratories, Inc., Maspeth, N. Y.
C. Hazleton Laboratories, Inc., Falls Church, Va.
D. Industrial Bio-Test Laboratories, Northbrook, Ill.
E. Syracuse University Research Corp., Syracuse, N. Y.
F. Institute of Paper Chemistry, Appleton, Wis.
G. Wisconsin Alumni Research Foundation, Madison, Wis.
H. Arthur D. Little, Inc., Cambridge, Mass.
I. Other private and/or institutional research organizations.

Note: No prejudice, whatever, is inferred against any such, or similar, organizations not specifically named above.

OUR SPECIAL RESPONSIBILITIES IN THIS FIELD

By the word "our" I mean to include, first, all TAPPI members whose specific duties and/or interests are involved, and, second, everybody else whose special duties and responsibilities (now, or in the future) are (or will be) connected with any paper industry program.

First of all, we need to have a broad knowledge of what is, and will be, involved. Then, we cannot afford to allow our emotions to submerge our logical thinking in the matter. And finally, we must do our best in cooperating with our associates and superiors in particular—if our duties are involved—we should study the specific releases to member paper companies from NPA and APPA, regarding raw materials, which are dated Oct. 2, 1959, and Oct. 9, 1959.

In the latter connection, the significantly important thing is the final form of a raw material (or of its residues, etc., if any) in the paper when it touches the food, not the initial form of that raw material when it was applied. For example, elemental chlorine is generally regarded as a toxic material under certain circumstances (even though its presence—as active chlorine in typical potable municipal drinking water at certain low concentrations—is generally regarded as safe by municipal and other responsible public health authorities). But because free chlorine simply never is still present in any paper coming off a paper machine, there is no point in asking for FDA approval for the use of chlorine in pulp and papermaking. Similar considerations apply in the case of numerous other pulp and paper industry raw materials. But, possibly, there might be cases where the interaction of some safe raw materials might result in reaction end-products that should be "cleared" with FDA.

Many people—including many of us that are present tonight—are apt to become confused when such matters as health, and public health statutes, are either allegedly or actually involved. In the case of food packaging, however, it is important to remember that the Food Additives Amendment of 1958, apparently, applies only to foods; apparently, said Amendment does not apply to drugs and cosmetics; and certainly it cannot possibly apply to any materials—including any packaging materials—not cov-

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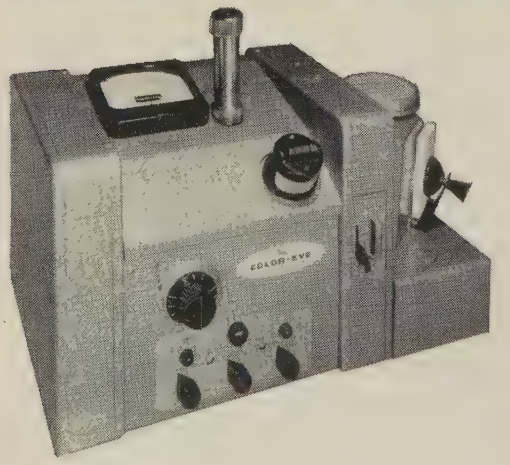

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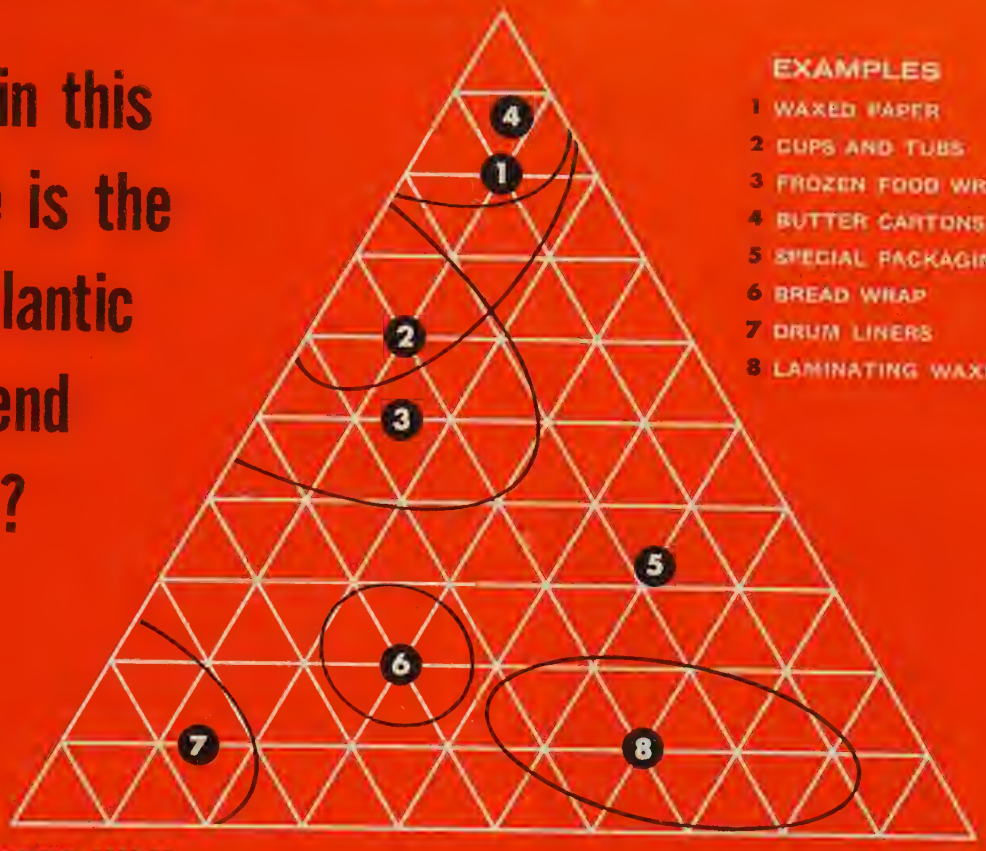

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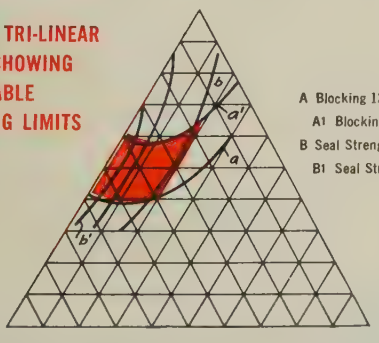
ADDITIVE

Somewhere in the tri-linear chart above is a composition of high-quality waxes just right for your operations. One of the many ways in which your Atlantic Sales Engineer can serve you is to pinpoint this position after he has made a thorough study of your individual requirements.

First, he determines the proper types of waxes for protection, convenience and sales appeal in the material you convert. Precise knowledge of the values he can ascribe to each component, including any required additives, means he can locate accurately on a chart the blend composition suitable for any end use in your plant.

As an example, wax coated wrappings for frozen foods must furnish good blocking resistance for easy handling and stacking . . . strong heat seal to retain freshness and flavor . . . low moisture vapor permeability . . . and attractive high-gloss appearance. The characteristics of each component in such a coating would be plotted on the chart to form the envelope-shaped area indicated here in red. Any proportion of the waxes within this area may be satisfactory for frozen foods packaging.

TYPICAL TRI-LINEAR CHART SHOWING ACCEPTABLE BLENDING LIMITS



But that's only half the task. Converting equipment, methods and the packaging material used vary widely throughout the industry. It therefore requires the experience and technical knowledge of the Atlantic Sales Engineer . . . backed if necessary by Atlantic's extensive research facilities . . . to discover within the general area a composition best suited to each individual use. This basic method of exact blend determination can be applied to any field where wax blends are used.

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The following is extremely important to us as chemical analysts, when actual, or alleged, food additives are involved: When the legally or otherwise imposed tolerance is absolutely zero, the available analytical methods *today* may not show any minute traces whatever in the final product, meaning that the use of a certain specific "chemical" earlier in the process is apparently permissible; but next year, or ten years from now, a new and more sensitive analytical method may show traces of said specific "chemical" in the final product, thus then "outlawing" the further use of the concerned "chemical" for the purpose concerned.

Another serious problem for us as chemical analysts is the amount of effort that we may have to devote to making transfer tests on papers and paperboards, along the lines outlined by FDA, particularly as referred to in the recent paper by Arthur A. Checchi, referred to above. The need for more sensitive, dependable analytical methods is most critical.

PROGNOSIS FOR THE FUTURE

When a certain administrative official of FDA spoke before the above identified M.I.T. Class in Food Technology in June, 1959, he ended his talk with approximately the following: "If you are confronted with a 'difficult' law, there are three alternatives: (1) Congress can change it; (2) the Court can change it; or, (3) you can comply."

Presented at the meeting of the Gulf Coast Section, Technical Association of the Pulp and Paper Industry, at the Battle House Hotel, Mobile, Alab., on Dec. 11, 1959

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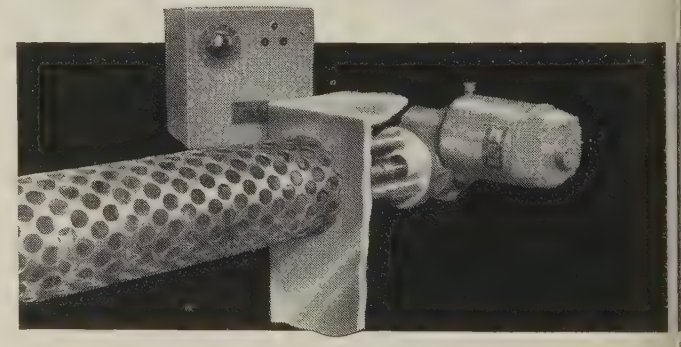

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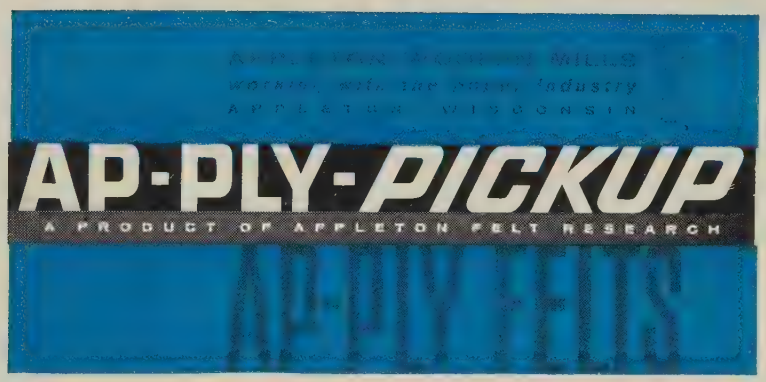

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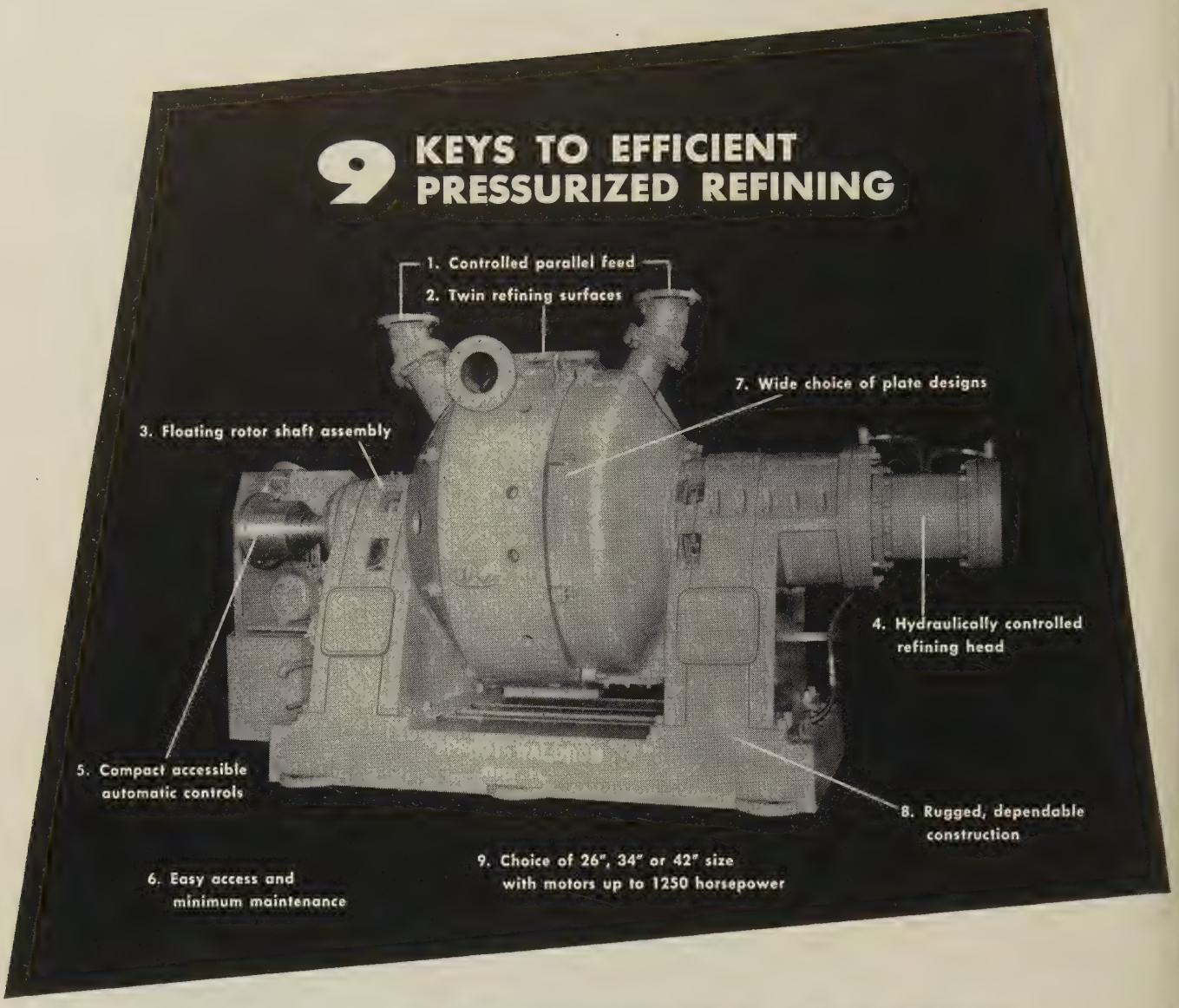
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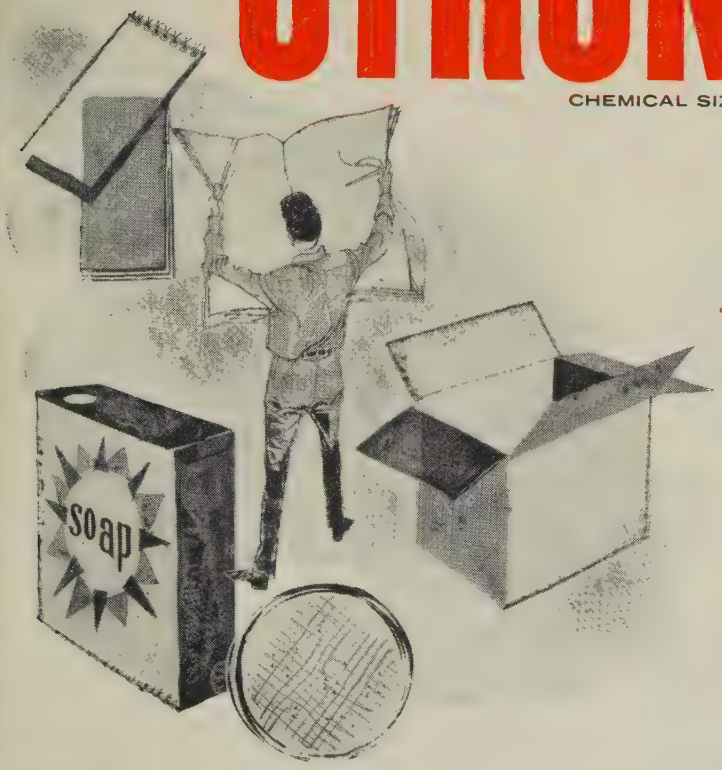

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1959 has been a paradoxical year. With the public press reporting good business for the country as a whole, it has taken a discerning student to discover that the part of the economy relating to capital goods has only bettered to a modest degree. This fact, plus the low carry-over of orders from 1958, caused us to have an unhappy year financially.

However, with the modest but positive increase in business in the U.S.A. and the substantial increase in orders for most of our overseas areas, we look forward with confidence to 1960. Improvement of efficiencies in all internal departments has also been positive and is continuing both as to costs and the time it takes to do things. This naturally will be beneficial to our customers.

SANITATION—Once again this segment of our operations has played a dominant role in our worldwide business during the past year. Orders were placed with us and our subsidiaries to serve larger centers of population such as Trenton, New Jersey; New York City; Bagdad, Iraq; Chicago, Illinois; and Elizabethville in the Belgian Congo, as well as for smaller municipalities from Pierrefond, Ontario to Albertville, Alabama and from the Villa Carmen housing development in Puerto Rico to Harlingen, Texas. Two large U.S. corporations also will install D-O equipment in treatment plants constructed for a research laboratory in New York State and a West Virginia townsite.

Further proof of widespread acceptance of our newer developments is installation of a SpiroVortex system at Checotah, Oklahoma and of CompleTreator units at industrial plants, recreation centers, housing developments and military sites in the United States. Abroad, our Dutch subsidiary will install one of these units in a concrete rather than steel tank, and the first CompleTreator unit has been purchased in the Philippines. The latter, for a large refinery, represents the first export order for our manufacturing representative in Malaya.

New applications of proven equipment include use of the ODS pump for handling conditioning lime for sewage sludge filtration and fabrication of a plastic sludge filter. Also, development work continues on additional applications of our industrial equipment in the sanitary field.

SUGAR—Orders from new and expanding cane sugar factories in India for RapiDorr Clarifiers and Oliver-Campbell Filters contributed significantly to the business of our subsidiary in that country. And in other locations around the world—Mexico, British Honduras, Egypt, Mozambique and Swaziland, for example—these two units continued to be accepted as standard in the industry. In beet sugar, a British producer purchased First Carbonation Thickeners and Vacuum Filters for two new mills; and two Italian mills will use a total of four Vacuum Filters.

PETROLEUM—A variation of the initial application of the DorrClone Desanding System in fresh water treatment is the desanding of salt water used to recharge wells for secondary oil recovery at a West Coast location. Outside the U.S. our Japanese representative is fabricating filters for use in a catalytic cracking process and our British company will supply oil-water separators for both British and Brazilian refineries. Also of interest is equipment for a pilot plant to recover oil from tar sands.

PROJECT ENGINEERING—This phase of our operations was also particularly active in 1959 as design of phosphoric acid plants to be located in Idaho, Great Britain and Ireland and a phosphoric acid and triple superphosphate installation for Brazil was commenced. Also in the fertilizer field was a contract for process modification of a large British plant.

Late in the year, work was started on design of a large limestone crushing plant for which D-O will also purchase equipment and other materials. Our French subsidiary and domestic company working jointly virtually completed design of a large Yugoslavian copper concentrator. Two smaller but nonetheless significant projects—both including laboratory testing and feasibility studies—involved production of a limestone substitute to be used for self-fluxing sinter in blast furnace operation and the recovery of nickel from waste pickle liquors.

WATER—Conventional pre-treatment equipment will be utilized at D-O supplied water plants for a new steel mill in India, a military camp in Iraq and the municipalities of Wilmington, Delaware; New Castle, Pennsylvania; Saida in Lebanon; and Maracaibo and Naiquata, Venezuela. Next year new PeriFilter systems will go into operation at New Oxford and South Pittsburgh, Pennsylvania and in 1959 a large system with two Hydro-Treator mechanisms went on line treating Midland, Michigan's water supply.

FLUOSOLIDS SYSTEMS—One of the new applications of the FluoSolids System this year has been preheating coarse shale in a special three-compartment reactor to produce light-weight aggregate. A second is drying iron ore before magnetic separation and concentration. Currently, the iron ore industry appears to have other drying applications ideally suited to fluidization. FluoSolids roasters are also under construction for a Canadian smelter where only partial sulfur removal is required.

Projects involving already proven applications have included in 1959 a pyrite roaster for a sulfuric acid plant on Formosa, three systems marketed by our Italian subsidiary for roasting pyrite to produce sulfur dioxide for pulp mill bisulfite cooking liquor and for a large zinc roasting installation in Yugoslavia, a large coal dryer for a Western producer, a copper-cobalt roaster in the Belgian Congo and detergent dryers in the United States.

METALLURGICAL—As in past years our work in this area has involved virtually all of the metallic minerals from gold to iron ore. The latter field has been among the most active with purchase of Sizers and Filters for Canadian washing plants, DorrClone classifiers for desliming prior to flotation and Thickeners, Filters and Pumps for recovery of blast furnace flue dust.

PULP AND PAPER—A new tool for this industry is a novel pressure washing filter. Installation of the first U.S. manufactured unit is being made in a Southeastern pulp mill. Ideally suited to brownstock washing, the unit has a number of advantages from both the operating and installation standpoints.

Recausticizing systems including process flowsheet innovations, such as precoat operation of the Lime Mud Filter and application of a two-compartment White Liquor Clarifier with special feedwell, were purchased for new or expanding mills in Canada, India, the United States, Portugal, Japan, Yugoslavia, Scandinavia and the Philippines.

Around the world the American Saveall continued to be recognized as the major means of recovering paper machine fibers from white water and in the U.S. the largest bleach tower designed to date will soon go into operation. An unusual and interesting application of the Merco Centrifuge is recovery of by-product material used as a drill mud dispersant at a West Coast mill.

CHEMICAL—During the year applications of the Plastic Filter, first introduced in mid-'58, were broadened to include a variety of fine chemical and pharmaceutical separations and washing of acid leach liquors from metallurgical pulps. Also in the realm of new applications is dewatering of various plastics using the Mercone Screening Centrifuge.

As usual, our standard line of equipment played a major role in a great number of Italian chemical plant expansions and in large new or expanded facilities in France, Germany, Spain, Mexico and here at home.

FOOD PRODUCTS—Successful application in 1958 of the Mercone-Precoat Filter combination for apple juice processing has resulted in orders from two West Coast packers for similar stations.

In starch, several domestic and overseas producers will employ D-O centrifugals of virtually every design for current modernization and process modification projects. Our Dutch subsidiary has put into operation three corn and potato starch washing and processing plants in Germany and Finland.

NON-METALLICS—A new clay and sand plant in Idaho will utilize considerable D-O sedimentation, filtration and centrifugal classification equipment and a Pennsylvania limestone producer has purchased Classifiers and a large Thickener for installation in an earthen basin. Also in the clay industry, the longest Oliver drum filters ever fabricated—24 feet in length—will be shipped early next year to a Southern producer.

We have noticed with much concern the apparent growing tendency of many users of services and equipment to buy almost exclusively on price rather than on demonstrated product quality and the essential engineering services made available to them during planning and after installation. This is not a new subject—and one on which there has been much forceful editorial comment in the technical press.

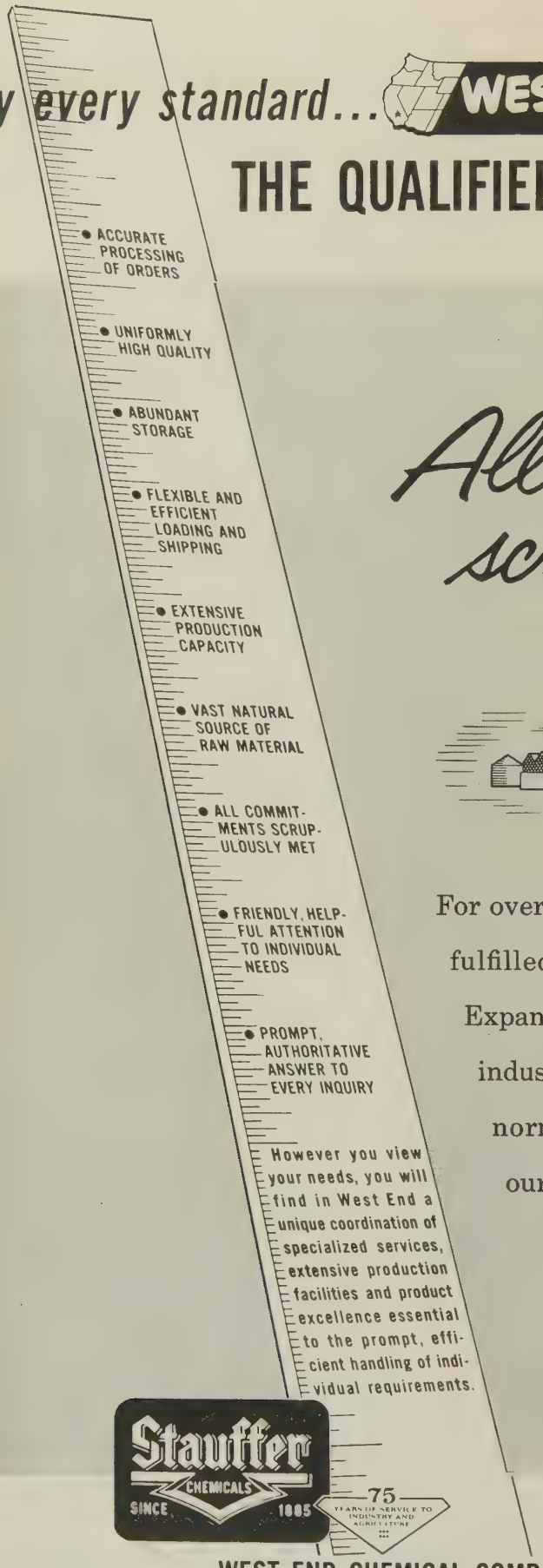
This practice confronts the supplier with the practical short range choice of sacrificing product quality and needed engineering, losing the business, or taking it at a loss. For our own part, we do not propose to fall into this trap which, economic considerations aside, can only result in arresting technological advance and which could have a profound effect on the leadership of the free world in things technical.

J. D. Hitch, Jr.
Chairman of the Board

December 1, 1959

RapiDorr, SpiroVortex, CompleTreator, DorrClone, Hydro-Treator, FluoSolids, Merco, Mercone, T.M. Reg. U.S. Pat. Off.

By every standard... WEST END MEASURES UP AS THE QUALIFIED SUPPLIER OF SALT CAKE


• ACCURATE PROCESSING OF ORDERS
• UNIFORMLY HIGH QUALITY
• ABUNDANT STORAGE
• FLEXIBLE AND EFFICIENT LOADING AND SHIPPING
• EXTENSIVE PRODUCTION CAPACITY
• VAST NATURAL SOURCE OF RAW MATERIAL
• ALL COMMITMENTS SCRUPULOUSLY MET
• FRIENDLY, HELPFUL ATTENTION TO INDIVIDUAL NEEDS
• PROMPT, AUTHORITATIVE ANSWER TO EVERY INQUIRY
However you view your needs, you will find in West End a unique coordination of specialized services, extensive production facilities and product excellence essential to the prompt, efficient handling of individual requirements.

All commitments scrupulously met

1925 1960

For over 35 years, West End has unconditionally fulfilled every commitment it has ever made. Expanding along with the chemical processing industry, West End meets the ever-increasing normal and unanticipated requirements of our customers with equal ease and dispatch.

Stauffer CHEMICALS SINCE 1885 — 75 YEARS OF SERVICE TO INDUSTRY AND AGRICULTURE

WEST END CHEMICAL COMPANY • DIVISION OF STAUFFER CHEMICAL COMPANY

1956 WEBSTER STREET, OAKLAND 12, CALIF • PLANT: WESTEND, CALIF.

an Invitation to PAPER COATERS

to explore
the possible advantages,
both physical and economic,
of using animal bone glue
as the adhesive in paper coating

ANIMAL GLUE, once used as the adhesive in paper coatings, is back in the picture. Improved glue qualities and new coating formulations have shown definite advantages—substantiated by laboratory evaluation, a full-scale paper mill trial, and actual printing tests.

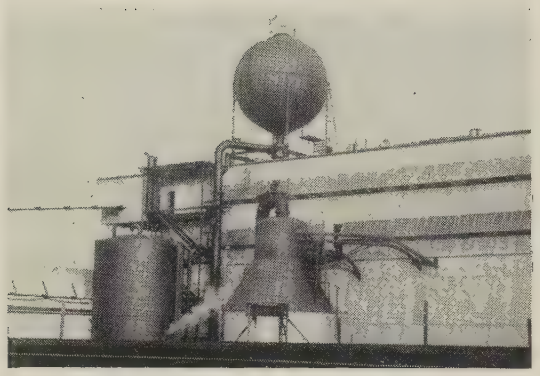
For a more detailed announcement of this development (printed on glue-coated paper), a technical bulletin describing the method used, or samples of DARLING's coating grade glue, write:

DARLING & COMPANY
GLUE DIVISION
4201 South Ashland Avenue • Chicago, Illinois

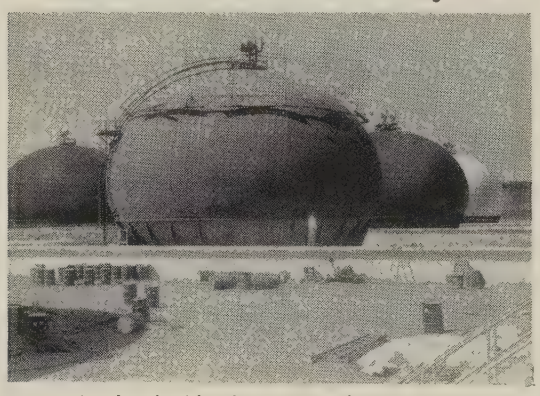
DARLING'S improved BONE GLUE

For corrosion protection, insulating, weather-proofing...

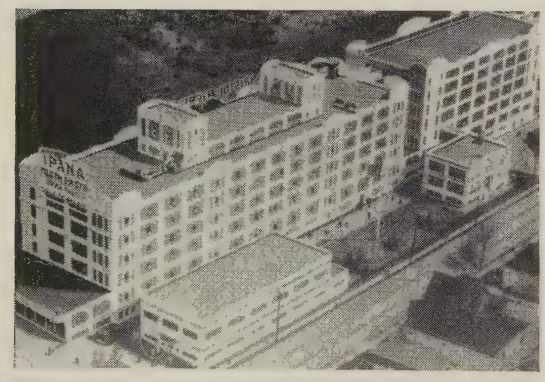
Industry selects ERKOTE® Mastics



Protected with Erkote 2X Corrosion Resisting Mastic



Insulated with Erkote 3X Insulating Mastic



Restored with Erkote White Mica Mastic

Distinguished names in American industry rely on ERKOTE Insulating and Corrosion Resisting Mastics to protect tanks, piping and structures against moisture, vapors and climatic conditions. They have discovered that maintenance costs go down when ERKOTE goes on. These *guaranteed* coatings provide a permanent protective barrier against destructive elements and guard your investment in expensive operating equipment.

Partial list of ERKOTE customers

Allied Chemical & Dye Corp.
American Cyanamid Co.
American Viscose Corp.

Beaunit Mills, Inc.
Bethlehem Steel Corp.
Bowaters Southern Paper Corp.
Bristol-Myers Co.

Cargill Inc.
Continental Can Co.
Crown-Zellerbach Co.

Delaware Power & Light Co.
Detroit-Edison Co.
Dow Chemical Co.
DuPont, E.I., de Nemours & Co.

Eastman Kodak Co.
Ethyl Corp.

Goodrich Co., B.F.
General Electric Co.
Gulf Oil Corp.

Hercules Powder Co.

Inland Steel Corp.
International Paper Co.

Kraft Foods Co., Inc.

Martin, Glenn L. Co.
Merck & Co., Inc.
Monsanto Chemical Co.

National Lead Co.
Niagara Mohawk Power Corp.

Olin-Mathieson Chemical Corp.
Owens-Illinois Glass Co.

Philadelphia Electric Co.
Powell River Co. Ltd.
Procter & Gamble Co.
Pure Oil Co.

Rayonier Inc.
Reynolds Metals Co., Inc.
Rohm & Haas Co.

St. Regis Paper Co.
Scott Paper Co.
Sears Roebuck & Co.
Simpson Paper Co.
Socony-Mobil Oil Co., Inc.
Standard Oil Co.
Sun Oil Co.

The Texas Co.

Union Bag-Camp Paper Corp.
United States Steel Corp.

West Virginia Pulp & Paper Co.
Weyerhaeuser Timber Co.
Wyandotte Chemicals Corp.

Complete responsibility for every application

We manufacture our own products, and we furnish our own trained crews and special equipment to apply them. When you select ERKOTE Mastics, you can depend on *one reliable source* for both the quality of the product and the workmanship.

ERKOTE can solve your protection problem

In refineries, in chemical plants, in paper mills . . . in every industry where insulation and corrosion protection are essential, ERKOTE Mastic coatings have a record of proven dependability. If you have a challenging surface protection problem, we will be happy to submit our recommendations.

ERKOTE®

TECHNICAL MASTICS

For use where paints fail

EARL PAINT CORPORATION • 240 Genesee Street • Utica, New York

Sales Offices: Chicago, Pensacola, Philadelphia, Seattle

Pump enough water in two minutes to make two thousand lbs. of paper!

To make a ton of paper, you need approximately 12,500 gallons of water.

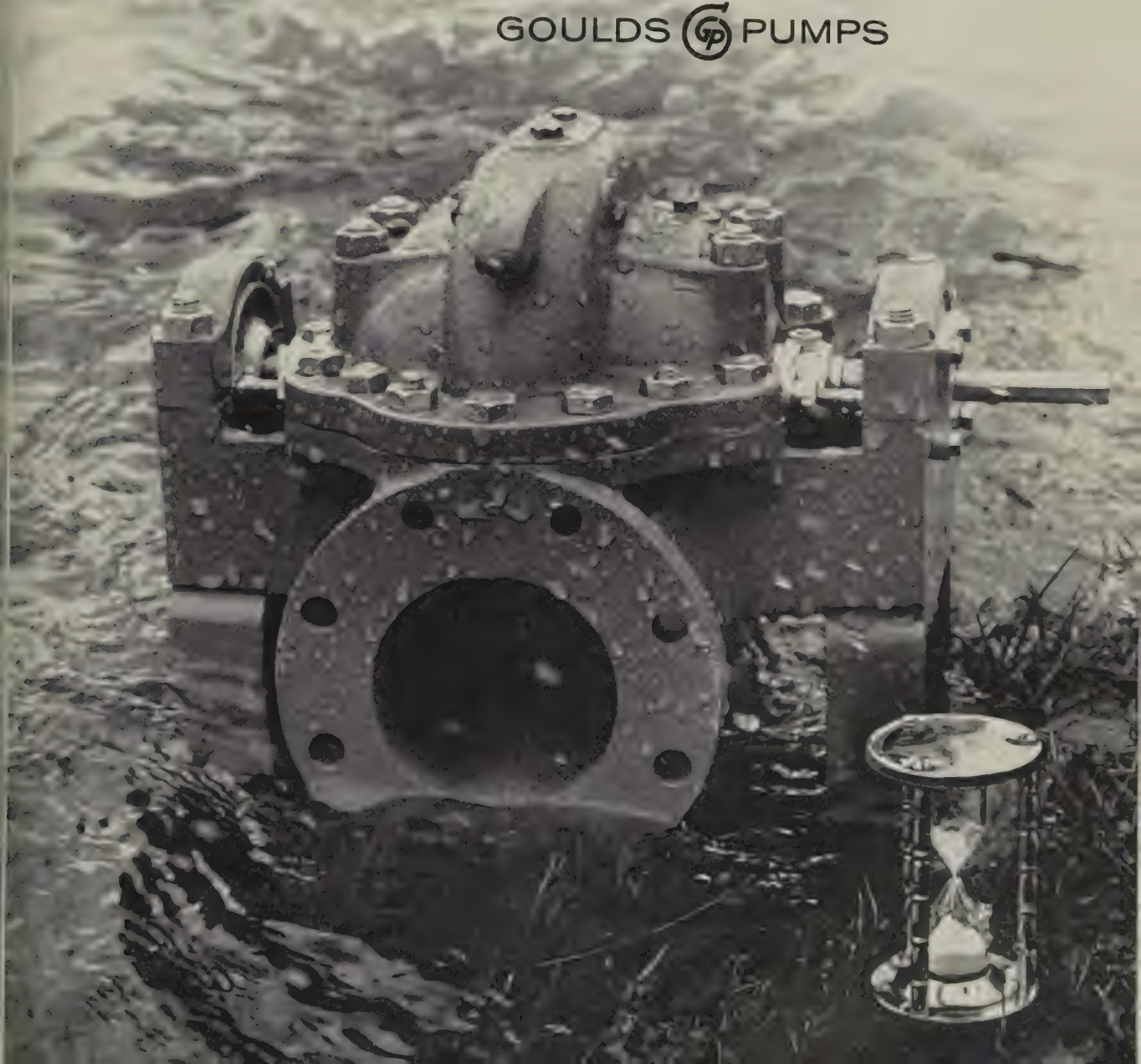
It would take just *two minutes* for a twelve-inch Goulds Fig. 3405 to supply it. This medium-size pump provides giant-size capacity: 6400 gallons per minute!

And to keep water flowing, you stock only a minimum of spare parts. Just three groups of parts fit thirty-three sizes of the 3405. All parts are interchangeable except casings, casing covers and impellers!

That means that for every three to five pumps of a given group size, you stock just one shaft, one set of bearings, sleeves, mechanical seals or packing. You can cut inventories as much as ninety-one percent, yet be fully covered for emergencies.

Heads go up to 425 feet. For more data, write to Goulds Pumps, Inc., Dept. TA-10, Seneca Falls, N. Y., for Bulletin 721.6.

GOULDS GP PUMPS



FOR IMMEDIATE DELIVERY

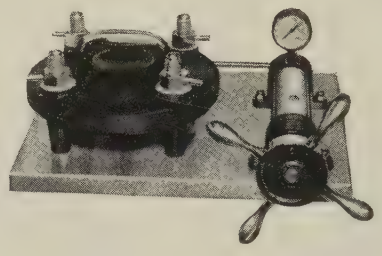
Tappi Standard

Pulp Testing and Sheet Making Apparatus

TAPPI SHEET MAKING MACHINE

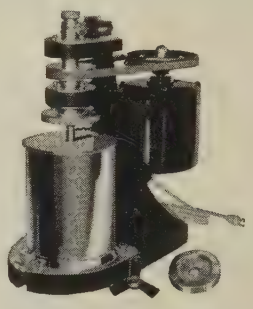
Bronze, brass and stainless construction throughout except couch roll which is heavily chrome plated. Drying Rings with gaskets and Drying Discs available but not illustrated. (T-205 m)

TAPPI PUMP & PRESS



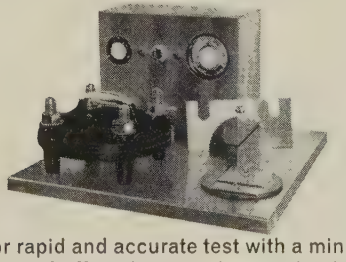
Mounted on 18-8 Stainless steel base. Press on base, or pump only may be purchased separately. (T-205 m)

TAPPI DISINTEGRATOR



Standard equipped with bronze chromium plated container bowl and stainless steel chuck, collet and shaft assembly. (T-205 m)

TAPPI PRESS WITH AUTOMATIC CONTROL

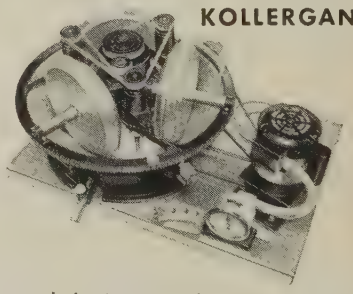


For rapid and accurate test with a minimum of effort. Base and control cabinet 18-8 stainless steel.

AUTOMATIC COUCHING DEVICE FOR SHEET MACHINE and FIBER LENGTH CLASSIFICATION GRID PLATE

(Not illustrated)

IMPROVED PRECISION KOLLERGANG



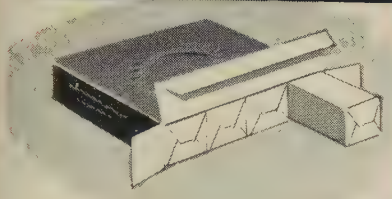
The only instrument that will give truly reproducible beating results. (T-225 sm)

This line of equipment features the ultimate in pulp testing and sheet making apparatus. Latest improved corrosion resistant material is used on all parts coming into contact with pulp solutions used in testing. The operational precision, durability and beautiful finish will command greater laboratory efficiency and cleanliness. All the Tappi Standard approved apparatus also bears the official seal and registered serial number of the Papermakers Association of Great Britain and Ireland.

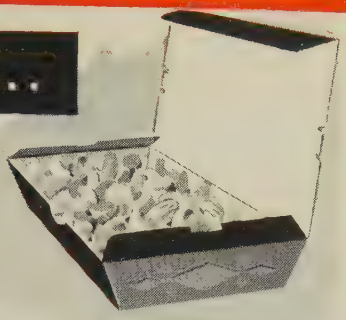
HERMANN MANUFACTURING CO.

LANCASTER, OHIO

IF YOU PACKAGE...



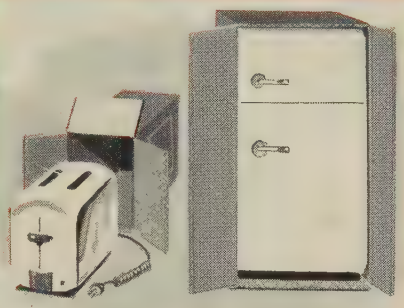
BUTTER, OLEO



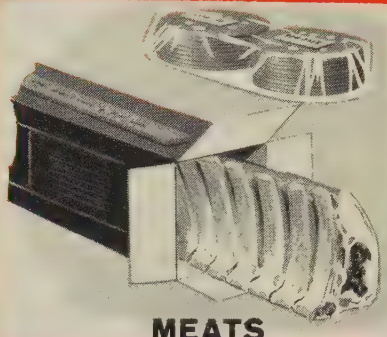
NUT MEATS

MILK, JUICE

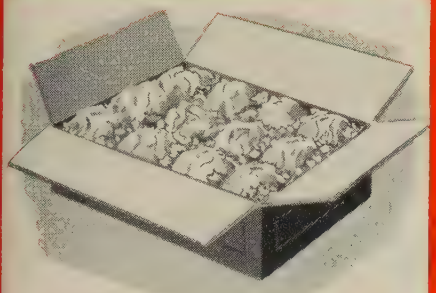
FURNITURE



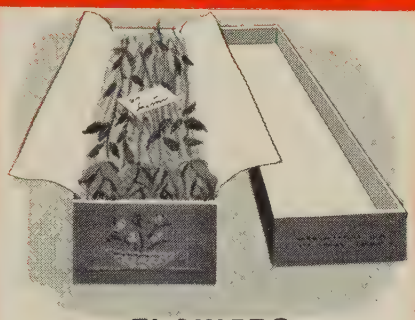
APPLIANCES



MEATS



FRESH VEGETABLES



FLOWERS

PASTRY

A-C® Polyethylene

CARTON COATINGS OFFER 7-WAY PROTECTION

- No more fiber scratch
- Improved scuff resistance
- Higher chemical resistance
- High grease resistance
- Better gloss
- Moisture resistance
- Reduced rub-off

If grease, moisture or fine finishes present packaging problems for your product, here's how you can pack and ship them safely *without recourse to costly carton liners:*

Incorporate A-C Polyethylene into your present carton-coating operation! It will eliminate fiber scratch and scuffing, give your cartons adequate resistance to grease, water and chemicals.

A-C Polyethylene can save you money, too. When you add it to ordinary wax coatings, *penetration is drastically reduced,* resulting in less wax consumption. For example, with straight paraffin, the normal coating weight is 7-8 lbs./1000 sq. ft. With addition of A-C Polyethylene, *it is frequently reduced to 3-5 lbs./1000 sq. ft. with superior protection!*

Write for literature, free samples. Free samples and technical data are now ready for your evaluation. Just write us at the address below. Or ask your local Semet-Solvay representative for a demonstration in your own plant. He can show you, right on your own equipment, how A-C Polyethylene can improve your cartons, save you money at the same time! *Contact us for the names of processors in your area who make up blends of A-C Polyethylene and wax.*

Allied Chemical

SEMET-SOLVAY PETROCHEMICAL DIVISION

Dept. 570-E, 40 Rector Street, New York 6, N. Y.

National Distribution • Warehousing in Principal Cities

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CABBLE WIRE COMPANY, INC.

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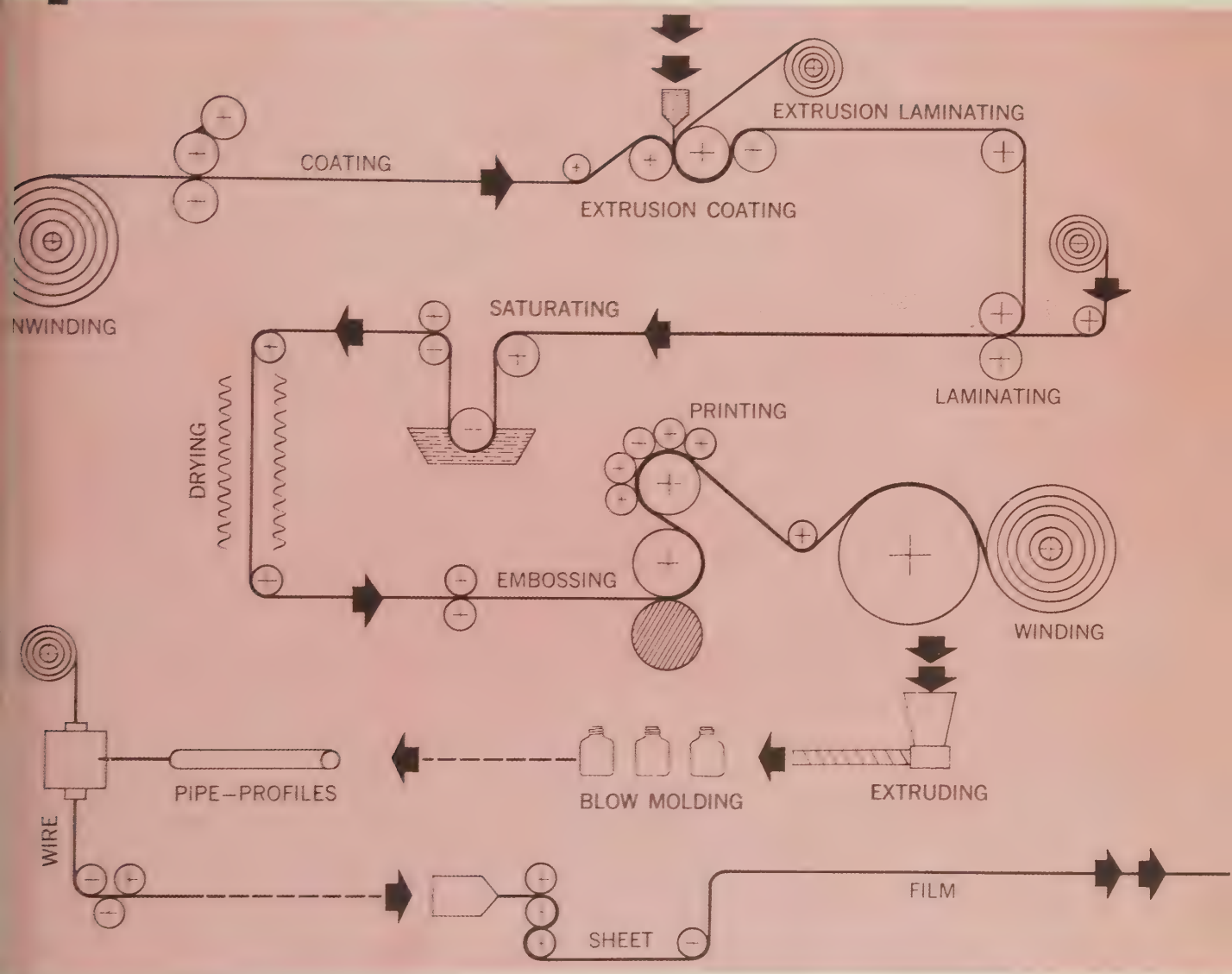
CABBLE FOURDRINIER WIRES

WALDRON-HARTIG DIVISION

LABORATORY FACILITIES AVAILABLE FOR EXPERIMENTAL AND PILOT PLANT STUDIES

WEB PROCESSES – DRYING – EXTRUSION



A DIVISION OF MIDLAND-ROSS CORPORATION

SCOPE OF SERVICES

In the past, the facilities of Waldron-Hartig pilot plant laboratories have been made available from time to time to customers for experimenting with new materials, processing methods and equipment, and for evaluating them for better product quality and cost reduction.

This arrangement has proved to be a good one, both for customers and for Waldron-Hartig. Customers have been able to determine the materials, methods, and equipment best suited to their present and future operations, and Waldron-Hartig engineers have had the opportunity to demonstrate the advantages of refinements and innovations in equipment in terms of the customers own production processes.

As a result, the facilities at New Brunswick and Mountainside pilot plant laboratories have been expanded, and are now available to customers and prospective customers on regular schedules, at a nominal fee. Both laboratories are fully instrumented, and are operated by a full-time technical staff. We welcome participation of your representatives in the test work.

The pilot plant in New Brunswick is designed to perform a variety of operations on webs of paper, plastics, textiles and foil in widths to 20" and at speeds to 2,000 FPM. A 24' long, straight pass, Ross dryer makes it possible to dry and cure a great variety of coatings and materials.

The pilot plant in Mountainside is equipped to compound, pelletize, devolatilize; to make blown film and water quenched film; to blow mold; to extrude pipe or profiles; to do wire covering, and to handle the many other processes associated with extrusion of thermoplastic materials.

ELIGIBILITY FOR SERVICES AND SCHEDULING

The Waldron-Hartig division laboratories are available to prospective customers for evaluating new products and determining the most suitable machinery for a processing operation.

Tests must be scheduled in advance due to the great demand for laboratory services. Any Waldron-Hartig sales engineer may be contacted to reserve time for laboratory tests. The maximum time that may be allocated for any customer's experiment is two days.

CHARGES.....A charge of $150.00/day is made.

A purchase order should be sent prior to the test. If an order for machinery is received within six months from the date of use of the laboratory, and the following credit allowance will be made:

orders of $10,000 or more—2 days test $300.00
orders of $ 5,000 or more—1 days test $150.00
orders of less than $5,000—no allowance

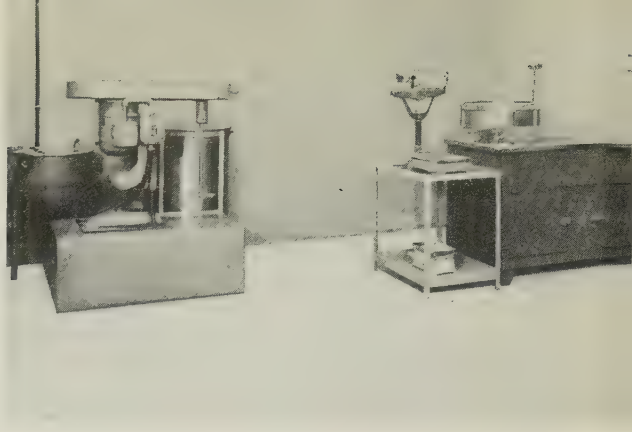
MATERIALS.....Materials for web processing must not exceed 20" in width, preferably wound on 3" I.D. cores and in rolls not exceeding 30" in diameter.

All materials required for the test are to be supplied by the customer. They should be sent—shipping charges prepaid—to the attention of the sales engineer contacted, and marked with the assigned laboratory test number. Make sure materials are sent in advance, as many times materials are lost or delayed in transit. Materials may be brought by the customer at the time of the test. Materials shipped direct from another supplier must be marked in the same manner. Materials can not be held for more than two weeks following a test; all raw and processed materials will be returned to the customer—charges collect—following the test unless other disposition is arranged.

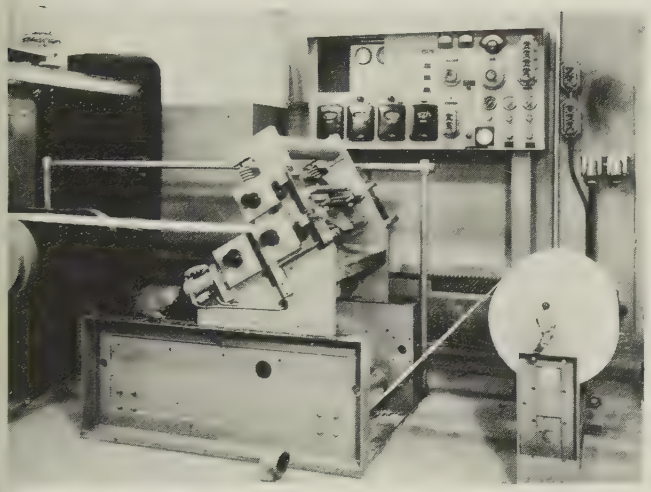
HOURS.....Regular hours are from 9:00 AM to 12:00 noon and from 1:00 PM to 4:00 PM during weekdays.

REPORTS.....Information gained is recorded on a standard laboratory report form. Data and samples and general test conclusion will be furnished the customer. All reports are held strictly confidential.

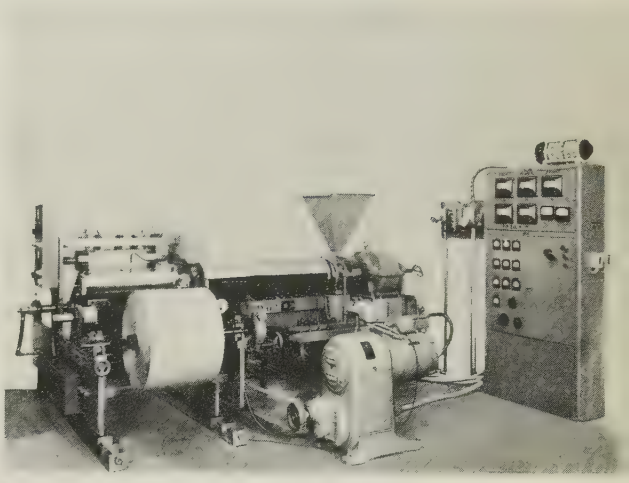
TYPICAL VIEWS OF THE LABORATORIES



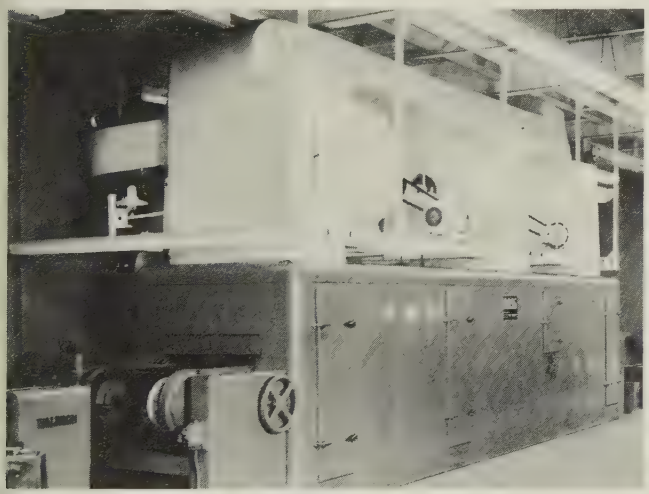
Cowles high speed disperser with low speed stirring adjustment. Double wall 25 gal. steam kettle, 50 gal. stainless steel mixing tank. Pound scale, gram balance.



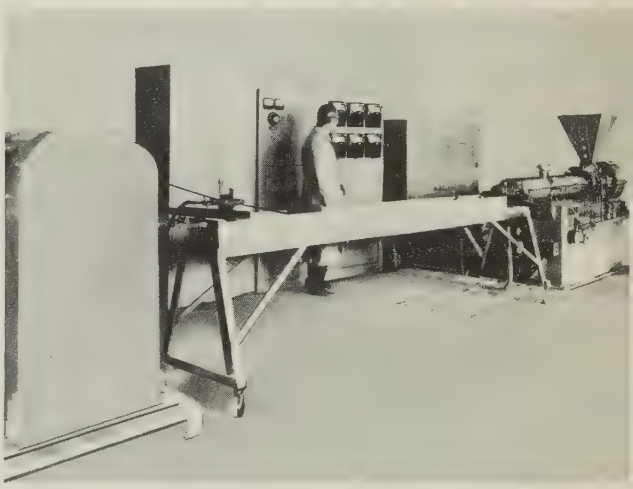
Reverse Roll coater, constant tension unwind. The coater base is designed to mount any of a variety of coating heads—either singly or in combination. Explosion proof drive and controls.



Extrusion coater/laminator/chill roll casting line.

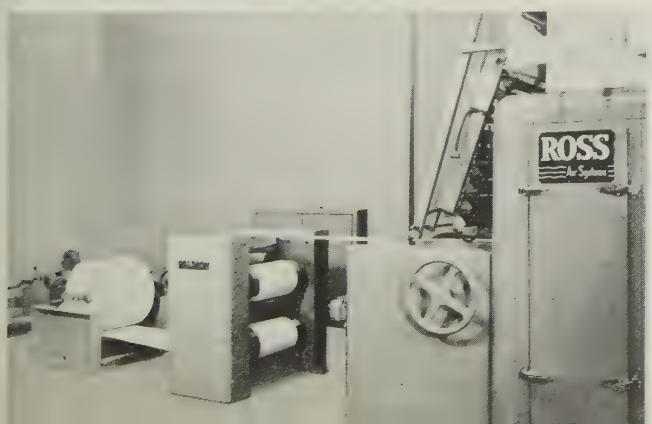


Three zone, 24′ Ross dryer equipped with pin tenter/stick conveyor.



2½″—24:1 L/D vented extruder, shown compounding and pelletizing. Many other assemblies are available.

Double drum cooling/pull rolls, guide, centershaft rewind. For low tensile strength materials, a special low tension centershaft winder can be mounted.



FACILITIES

The Waldron-Hartig Division Laboratories are in two locations—New Brunswick, N. J., and Mountainside, N. J. The laboratory in the Waldron plant in New Brunswick is primarily for web processing studies, and the laboratory in the Hartig plant in Mountainside is primarily for extrusion studies. Both have the necessary auxiliary equipment for handling associated processes. In addition, the use of facilities of the J. O. Ross Engineering Division laboratory, immediately adjacent to the Waldron plant, can be arranged for those applications found to require broader drying or curing facilities.

The following list of equipment, although not all-inclusive should serve to define the scope of testing facilities.

EQUIPMENT

WEB PROCESSING

Coaters ..... Microjet (air knife coater)
Reverse Roll/3 and 4 roll
Extrusion/18″ dia. chrome plated cooling rolls, mirror/buffed/commercial finish
Knife: over roll/over blanket/floating
Kiss/1 and 2 sides
Trailing Blade/Levelon

Impregnators Dip and Squeeze, Dip and S Wrap, Vacuum and Pressure — Steel/chilled iron/rubber rolls/or combinations

Printers ..... Gravure/direct and offset

Laminators .... Teflon and Silicone rubber rolls available

Embossers .... 20,000# hydraulic; shallow, medium and heavy engraved steel rolls for use with rubber rolls

EXTRUDING

Extruders ..... 2½″-24:1 L/D, vented barrel; 2½″-21:1 L/D — Screw Speeds to 200 rpm

Processing screws for both extruders include many optional types such as polyethylene, nylon, vinyl, double metering, blister, and special compounding. Masticating head attachment, and many die configurations for multiple strand extrusion, tubing, blow molding, blown tubing and wire and cable covering.

DRYING & HEATING

Dryers ..... 24′ long straight pass, horizontal Ross dryer with adjustable header positions and a range of air velocities of 2,000 to 10,000 FPM in 3 zones of individually controlled temperatures of 450°F to 500°F makes it possible to dry and cure a great variety of coatings and materials. The top or bottom headers can be used individually, and a flat plate or rubber belt conveyor used to support fragile materials. A stick conveyor can be used to speeds of 500 FPM. The conveyor can be converted to a pin tenter for textiles.

Heaters ..... 3 banks of Chromalox radiant heaters with 6 units each for a total of 20 KW are available, for fusing, heating, etc.

15 KW 20 gpm hot oil heat transfer system is available for heating rolls and coating pans.

In addition, our affiliated company specializes in, and has general air handling systems including:

Medium and high temperature batch ovens to 1400°F. (small and walk-in).

Direct-fired or indirect heated high velocity web dryer, 0 - 20,000 FPM air velocities, with floater section.

Web conditioning apparatus.

AUXILIARY EQUIPMENT

Unwind, constant tension
Centershaft winder, taper tension
Centershaft winder, light tension (½ hp dynamatic)
Centershaft winder, light tension (8 ft - lb. torque motor)
Web guide
Guided suction apron
Pelletizer, 4 blade
Take up, blown tubing
Capstans, wire and cable
Reels, wire and cable
Water circulating system, 75 gpm (with 50 gal temperature controlled reservoir)
Steam boiler, 15 hp at 15 psi
Centrifugal blower, 350 cfm, 4 psi
Air Compressor, 45 cfm, 100 psi
Rolls—large assortment of completely, interchangeable rolls — rubber, neoprene, and silicone rubber (durometers of 25 to 100) chrome plated, chilled iron, teflon coated, engraved (coating and embossing)
Vibrator screen
2 Clay slurry pumps, 61 gpm with variable speed motor input
Stainless steel pump, teflon lined
Stainless steel pump, neoprene impeller
2 vacuum pumps
Coating pans of all materials, shapes and sizes

INSTRUMENTS

Microscope 10x-10x, 10x-43x
Microscope, low power, 3 dimension
Pyrometers, 3—indicating (0°F-800°F)
Viscosimeter, Brookfield Model RVF
Viscosimeter, Zahn, #1 through #5 cups
Wet thickness gauges, 0-.004″, 0-.012″
Tachometers, hand, instantaneous electric, counter type
Comparator, bench — type, 0-.012″, .0001″ graduations
Indicator, dial, .0001″ graduations
Thermometers, complete range of mercury and dial types
Camera, Speed Graphic, 4x5
Hot Plates, 2
Watt meter, indicating, portable clamp-on
Watt meter, recording, Esterline Angus, 5-300 hp
Cylinders, volume, assorted
Gauges, pressure, bleed type Bourdon tube, ranges to 10,000 psi

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Observation of equipment operation ☐ Other ☐ (Describe)_____

GENERAL INFORMATION:

Is this operation being performed commercially at present? Yes ☐ No ☐
Is present operation inadequate? Yes ☐ No ☐
If inadequate, describe:_____

Will drying be required? Yes ☐ No ☐
If yes, temperatures required Zone 1_____ Zone 2_____ Zone 3_____
Raw Material: Amount and type such as web width (20" max.)_____
Thickness_____ Kind of Material_____ Weight_____
Core I. D._____ (3" preferred)_____ Quantity supplied_____
Requires Blending ☐ Mixing ☐ Comes Ready to Use ☐_____
Is any of the raw stock toxic? Yes ☐ No ☐ If yes, elaborate_____

How will it be shipped? ☐ Truck ☐ R. R. ☐ Brought, other_____

What are some of the final products specifications as in regard to this test? (For example ½ mil continuous film, high gloss, complete impregnation, surface smoothness, non-porosity, etc.)_____

Equipment set-up. It is customary for Waldron-Hartig to select the most appropriate equipment to produce the desired results. If specific equipment or a special set-up is desired please sketch on a plain sheet of paper and attach to this form.

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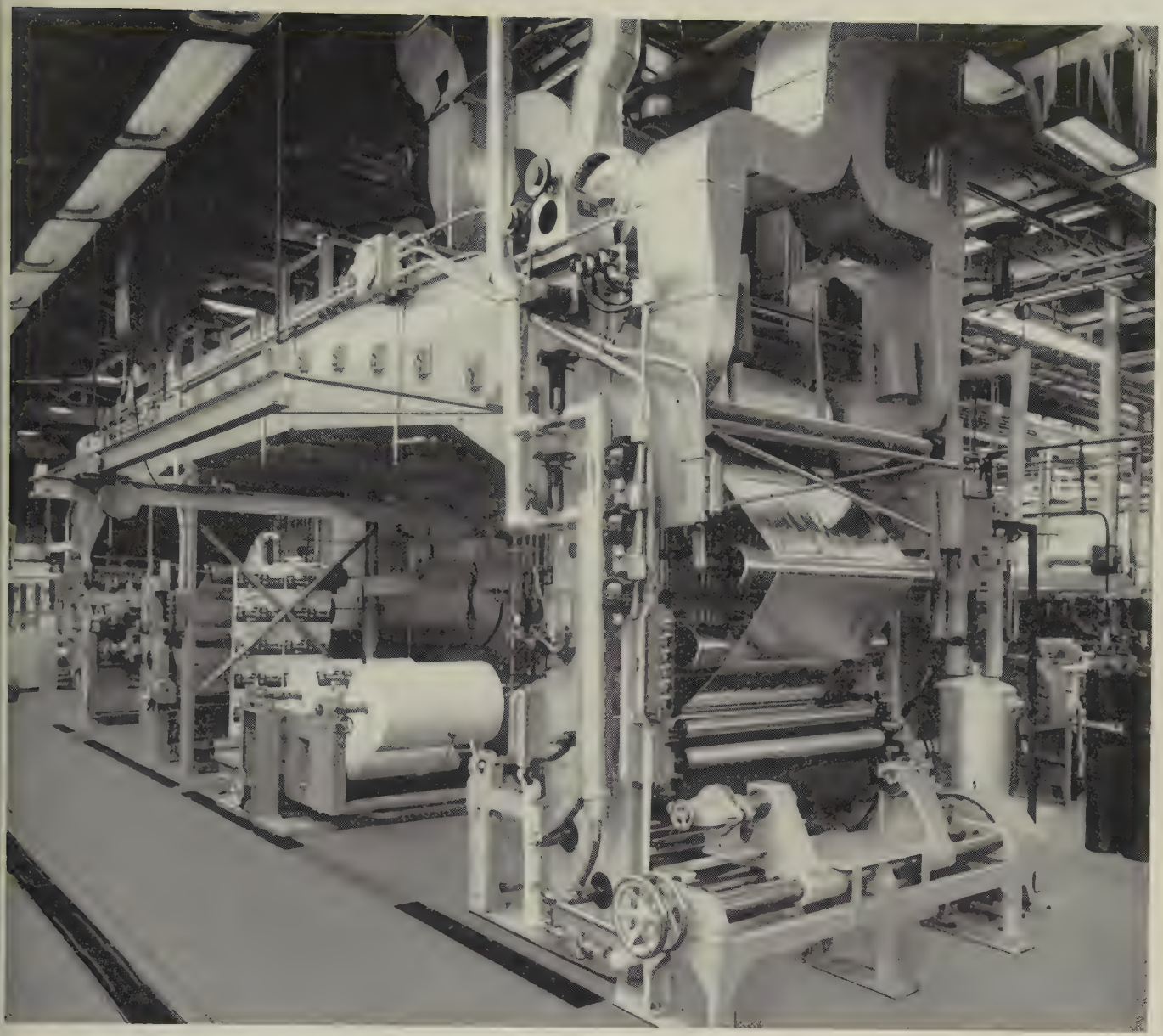

Inta-Roto GM-2000 Laminator Expands Western-Waxide Facilities

Above is an actual photo of the Inta-Roto GM-2000 Laminator in use at Western-Waxide Division, Crown Zellerbach Corporation, San Leandro, California, headquarters plant. David J. Benjamin, Vice-President of the company, had this to say about the GM-2000: "This machine is the finest of its kind for the production of high precision laminated and/or coated flexible packaging materials.

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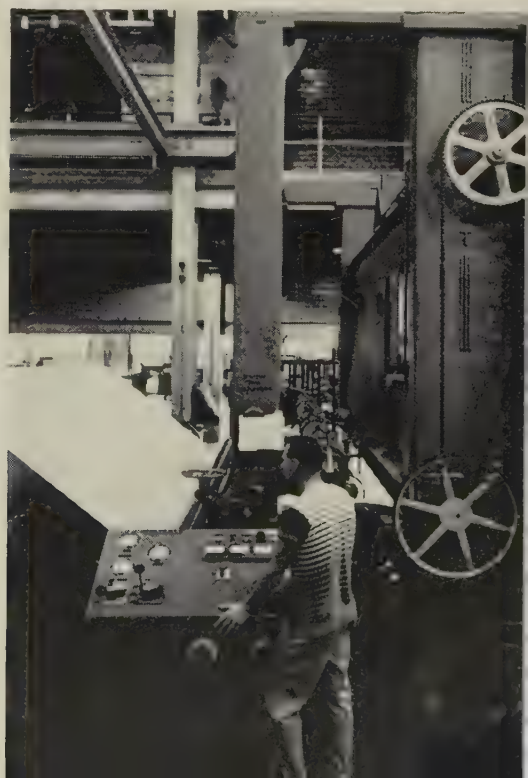

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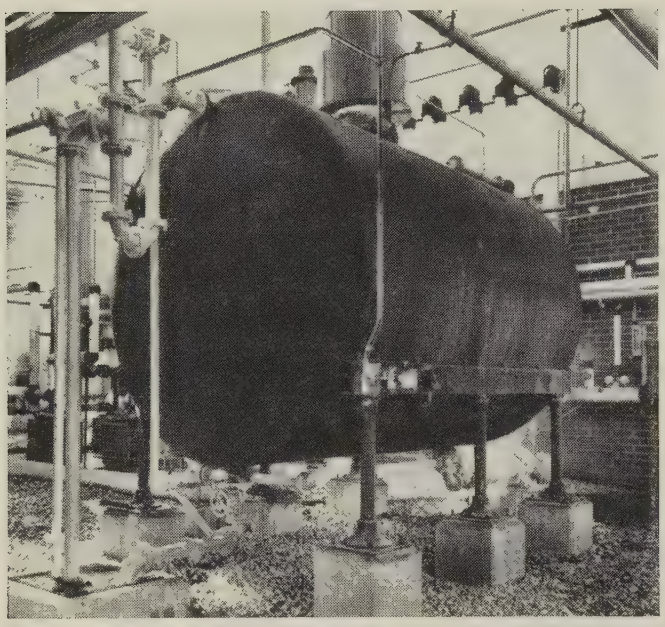
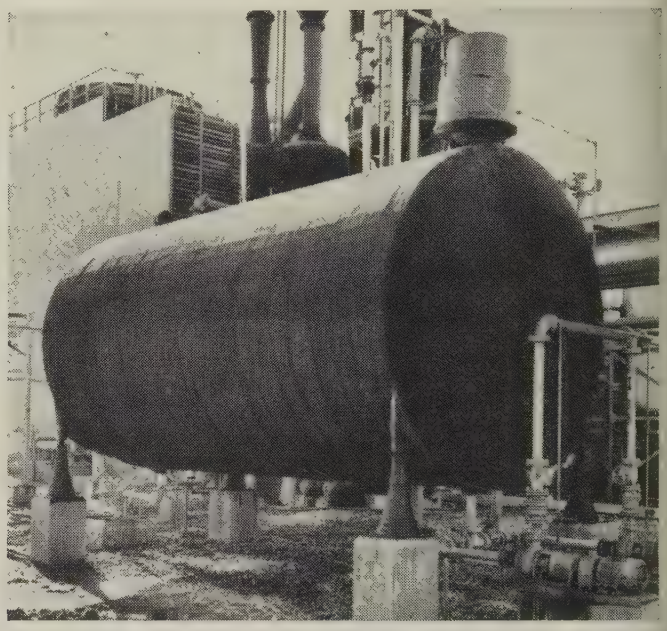


Waste liquors, highly corrosive, are accumulated in Glasteel at Marathon Southern in this 5000-gallon Glasteel Chemstor tank.



15,000-gallon storage tank of Glasteel construction keeps chlorine dioxide safe and pure until it's ready for use.

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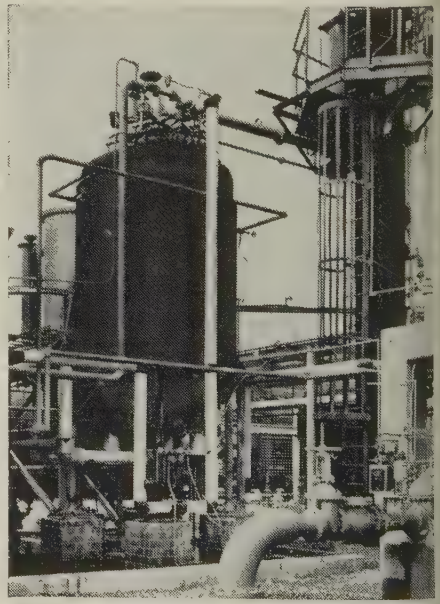

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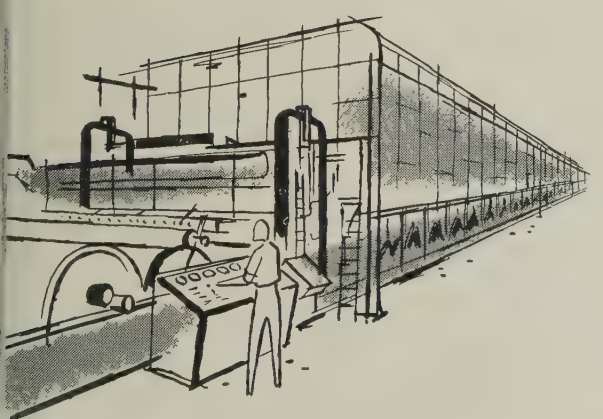

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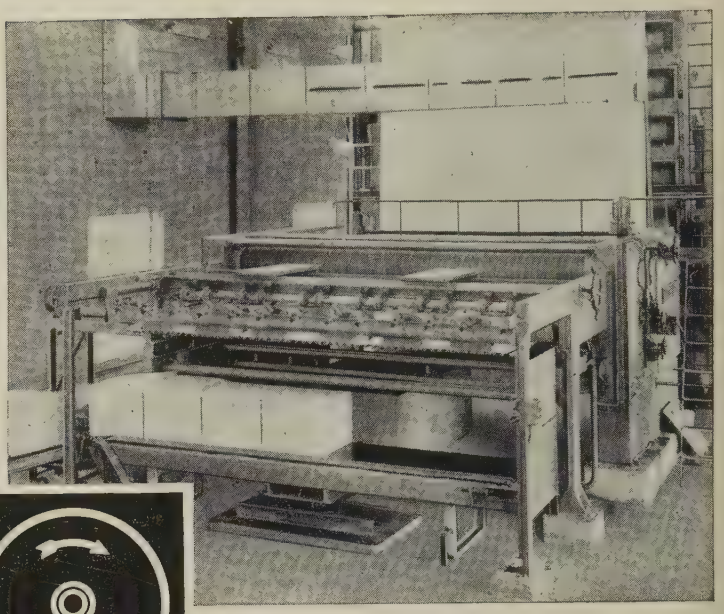

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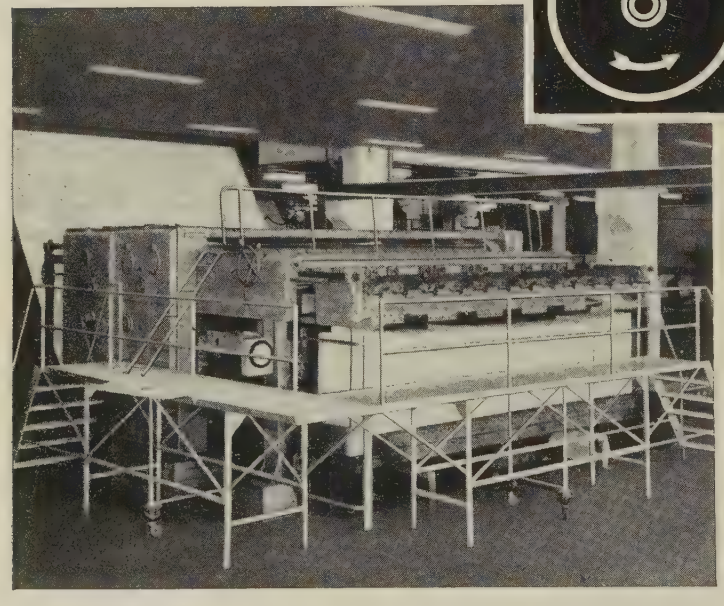

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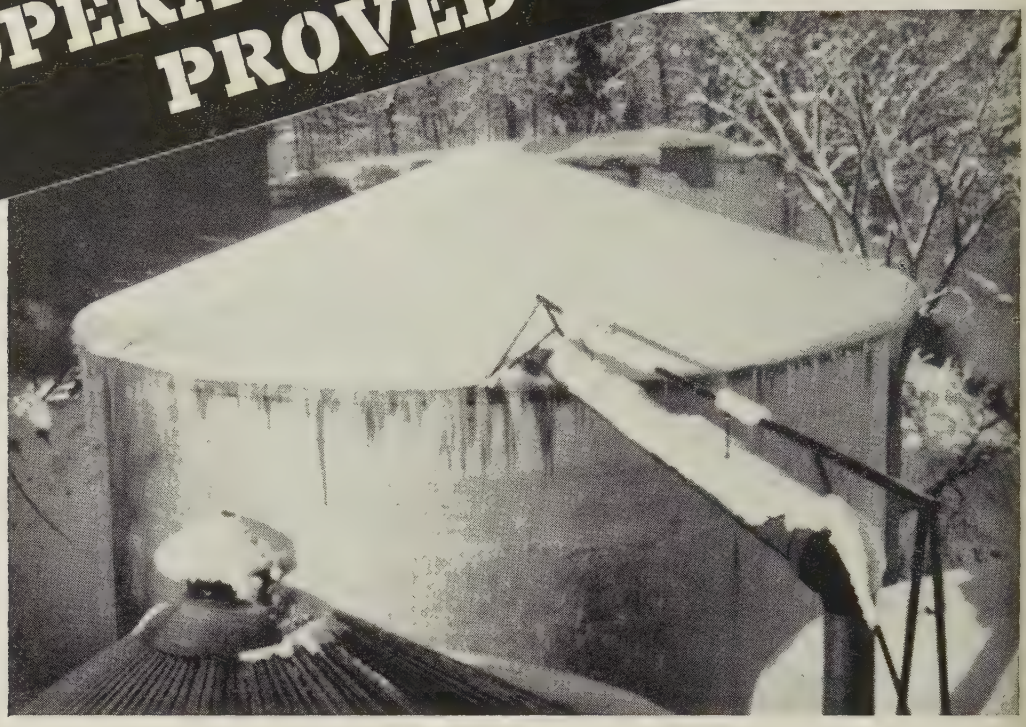

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The Juvenile Core in Conifers

BENSON H. PAUL

Juvenile wood comprised wide-ringed, low-density material of high longitudinal shrinkage near the pith. Narrow-ringed wood of the same species lacked these undesirable characteristics. Thus trees originating in closely stocked stands contained only negligible amounts of juvenile wood. The number of juvenile growth rings appears to be related to the size and position of the green crown. Thus its amount is subject to control by regulated stocking in forest management.

THE subject of juvenile or "core" wood—wood that often comprises several growth rings immediately surrounding the pith in coniferous stems—recently had been given a great deal of attention by wood technologists and others. One reason for this is the fact that the many second-growth, old-field stands and plantations, in which the southern pines and some other species are now predominant, produce relatively large amounts of this undesirable kind of wide-ringed wood in comparison with the amounts that were in present stands of old growth and second growth that originated under more restricted conditions.

More than 20 years ago Koehler (*2*) and Paul (*4*) found that 2 by 4's cut from the wide-ringed cores of old-field southern pines warped considerably because of high and unequal shrinkage along the grain. This high shrinkage occurred mostly in growth rings more than $^1/_4$ in. wide near the pith. Further studies revealed severe bowing in pieces cut from stems of plantation-grown slash pine about 15 yr. of age. The lengthwise shrinkage of the central core and of these young slash pine trees was up to 10 times as great as that of the outer wood.

For comparison with the plantation-grown trees, other slash pines of slower growth, with initial ring widths of about $^1/_8$ to $^1/_{10}$ in., were obtained from nearby thickets of mixed hardwoods and pines. Sticks cut in the same way and dried in the same manner as the plantation stock did not warp or twist. They remained straight, with the edges parallel to the line of a saw kerf cut longitudinally along the pith.

How can juvenile wood be defined? Is it wood near the pith that has wide rings, which cause excessive longitudinal shrinkage and warp? Or is it narrow-ringed wood near the pith that is well behaved from the standpoint of shrinkage and warping? Or can it be both?

According to Zobel, Webb, and Henson (*6*), there always are a few rings of low-density core wood at the center of a tree, regardless of ring width. In reporting their investigations, however, they remarked that no intermediate or suppressed individuals were sampled. Thus, the very trees most likely to contain little or no juvenile wood were omitted from examination. In their discussion, these authors observed that greater variation in the number of juvenile rings was noted than had been recorded in some earlier publications.

It undoubtedly is true that early suppression or training of young trees is necessary in order to reduce the core wood to a negligible amount. Keeping rings to $^1/_8$ or $^1/_{10}$ in. in width for the first 10 yr. possibly would be sufficient to accomplish this, but a longer training period is probably desirable in order to extend the narrow-ringed core to a greater height. This is all a matter of forest management. In planations on clear-cut areas and in thinly seeded old fields, a fairly large core of lower density wood around the pith may be expected. The size of this core appears to be determined by the rapidity with which a stand closes—that is, by the early or late dying of side branches and the resultant upward movement of the functioning crown.

It seems reasonable to relate the amount of core wood to the magnitude of the green crown. It is formed when green foliage is fairly close to the stem area of growth—that is, at the same height along the bole. This is in agreement with Iablokoff's "bud hormone" action (*1*) and Trendelenburg's "crown-formed wood" (*5*). By high pruning of open-growth longleaf pine trees, Marts (*3*) caused an abrupt change from wood of the juvenile type to that having mature characteristics.

The formation of core wood in sufficient amounts to cause warping of lumber is not an intrinsic trait of tree growth (nature did not intend that it should be). Rather, the forester through application of silvicultural principles should so manage and reproduce a forest that the amount of core wood will be negligible. Unfortunately, this calls for a different management system than many now under way.

For many years forest practices in continental Europe have been carried on in such a manner that core wood was practically absent—or at least it occurred only in a minor degree. Forestry practices, however, were aimed at reproduction of the stand as the result of several partial cuttings, extending sometimes over as many as 50 to 60 yr., or by extremely close planting. Dr. W. Knigge† has told this author that the European forest practices are aimed not exclusively at preventing juvenile wood but also at attaining other advantages, such as the improved natural pruning that results from small side branches, smaller knots, fewer black knots, a more uniform product, and cheaper sawmilling.

Restriction of core wood development is important if we are to produce trees suitable for other and more valuable uses than fiber products, or if we wish to avoid the necessity of separating core wood from lumber in the course of manufacture, as is being done with radiata pine in New Zealand. Core wood is all too easily grown.

BENSON H. PAUL, Forest Products Technologist, Forest Products Laboratory, Forest Service, U. S. Department of Agriculture.
Maintained at Madison, Wis., in cooperation with the University of Wisconsin.

† Dr. Wolfgang Knigge, Institut für Forsthenutzung der Universität, Göttingen.

Preventing it or restricting it to a negligible amount challenges the skill of the silverculturist.

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Received Nov. 25, 1959

The Transverse Tensile Strength of Clay-Starch Coatings

ARNOLD C. EAMES

In order to approach a more fundamental understanding of the strength of pigment coatings, the internal transverse tensile strength (TTS) of clay-starch coatings was studied as a function of adhesive distribution. TTS was measured by a new method based on the familiar principle of cementing flat, solid surfaces to either side of the specimen and pulling the specimen apart in a tensile tester. The new TTS method differs from older methods (1) in the adhesive used, (2) in the technique of mounting the specimen, and (3) in the manner of applying a uniform, transverse tensile stress. On porous substrates TTS increased with (1) decreasing pigment particle size, (2) increasing substrate pore size, (3) increasing adhesive content, and (4) lower rates of drying. Coat weight variations had little effect on TTS. When pigment particles could penetrate the substrate pores, a new zone of minimum TTS was found in the top third of the coating. The experimental results can be explained in terms of the final configuration and distribution of the starch. The mechanism of starch distribution is thought to involve primarily capillary competition for the vehicle between the substrate and the coating but is also influenced by the drying forces. The TTS results are not directly applicable to pick strength, but observations useful in pick strength analysis were made.

A NEW APPROACH TO COATING STRENGTH

As a step in the direction of more fundamental coating strength investigation, it was proposed to study the behavior of a simple clay-starch coating under the influence of a uniform tensile stress applied perpendicularly to the plane of the coating. This strength property, called transverse tensile strength (TTS), is defined as that tensile stress required to cause failure in a sheetlike specimen when a uniformly distributed stress is applied perpendicularly to the plane of the specimen. TTS has been selected because it appears to play a very significant, if not dominating, role in pick strength.

The substrate is to be considered only in terms of its contribution to the final properties of the coating, for example, by its effect on the loss of vehicle from the coating by penetration into the substrate. There are two reasons for this restriction: (1) Failure of coated paper under printing conditions usually occurs within the coating. (2) Failure under pure transverse tensile stress might occur in the substrate, but such a failure would be of minor significance to a quantitative understanding of coating strength.

Void volume distribution, stress concentrations, residual stresses, and particle alignment are believed to affect the distribution of TTS within a pigment coating; but their relative contributions to TTS are thought to be subordinate to, or dependent on, the adhesive distribution.

The two major objectives of this study were: (1) to develop a means of measuring the internal TTS of clay-starch coatings, and (2) to analyze the TTS behavior of clay-starch coatings as a possible function of adhesive distribution.

The experimental approach was (1) to vary certain independent variables known or believed to affect adhesive distribution and then (2) to determine the TTS, the location of failure, and the net adhesive ratio of the coating. The independent variables chosen were: (1) pigment particle size, (2) substrate pore size, (3) initial adhesive ratio, (4) method of drying, and (5) coat weight.

THE SYSTEM: DESCRIPTION OF MATERIALS

Clays

The three particle size fractions of "Lustra" coating clay used in this study were obtained from Southern Clays, Inc. Kraske (*1*) has described the clays in detail. Particle size distributions obtained by the centrifuge method and expressed in terms of equivalent spherical diameter (e.s.d.) are shown in Fig. 1 (*1*). The three clay fractions are identified by the code letters given in Fig. 1.

Starch

Because of its widespread use in the pigment coating of printing papers and its availability in sufficient quantity with uniform properties, Stayco M, a low-viscosity hydrochlorite-oxidized cornstarch, supplied by A. E. Staley Co., was chosen.

Arnold C. Eames, Graduate Student, The Institute of Paper Chemistry, Appleton, Wis.; present address, S. D. Warren Co., Westbrook, Me. Work performed at The Institute of Paper Chemistry.

The starch was slurried at 25% solids in distilled water and cooked in the Corn Industries viscometer, described by Kesler and Bechtel (*2*), at 96°C. for 30 min. After cooking, the starch was cooled to 50°C. and held at this temperature until it was added to the clay slip. In no case was the starch preparation held at 50°C. longer than 3 hr.

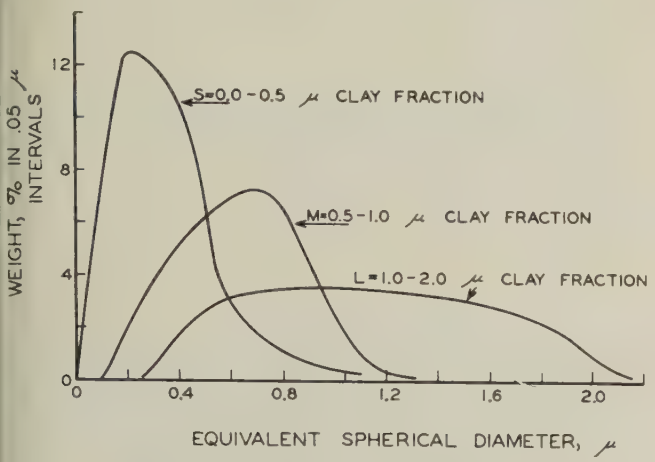


Fig. 1. Particle size distributions (1)

Characteristic values obtained during a starch cook are:

Temperature at start of swelling = 62°C.
Maximum torque during the cook = 260 g. cm. at 69°C.
Torque at 50°C., after cooking = 12 g. cm.

Substrates

Two classes of substrates were used:

1. Nonporous—aluminum foil, 0.006 in. thick.
2. Porous—Millipore Filter (MF), a synthetic sheet material made by Millipore Filter Corp. Three types of MF, types VC, HA, and AA, were used. They are identified by the code letters given in Table I describing their pore sizes and flow rates. MF is characterized by a smoothness comparable to cast-coated papers such as Kromekote, a refractive index of 1.5, a void fraction of 0.8, an inertness to water or dilute alkalies, and solubility in certain organic solvents, such as ethyl acetate which do not swell starch. The pores in MF are oriented perpendicular to the surface of the filter and thus provide essentially direct passages through it.

EXPERIMENTAL METHODS

Preparation of Coatings

A typical coating formula is:

Weight of solids, g.	*Component*		*Total weight, g.*
200	Clay, 1.0–2.0 μ at 99.4% solids	=	201
24	Starch at 25% solids	=	96
	NaOH, 1.0 *N*	=	9
	Quadrafos ($Na_6P_4O_{13}$), 1.0 *N*	=	6
	Water	=	95
224	at 55.0% solids		407

The clay is added to the solution of sodium hydroxide and Quadrafos (made by the Rumford Chemical Works) in distilled water under agitation with a Hamilton Beach stirrer. About 10% of the sodium hydroxide and water called for in the formula is withheld for later adjustment of the pH and solids content of the coating. When the clay slip has been stirred for at least 30 min. or until the clay is well dispersed, the required amount of starch is weighed into the clay slip. The starch preparation has been described above.

Table I. Pore Size Characteristics of Millipore Filter (*3*)

Coating code letter	*Millipore filter type*	*Pore size, μ*	*Pore diameter variations, μ*	*Rates of flow, cc./cm.²/min. at 25°C. and 70 cm. Hg*	
				Water	*Air*
V	HV	0.100	0.008	3.6	600
H	HA	0.45	0.02	72	9,600
A	AA	0.80	0.05	330	33,000

After the coating is thoroughly mixed, the pH is adjusted to 8.0 with 1.0 *N* NaOH. The coating is then passed twice through a hand-operated dispersion mill (Chicago Apparatus Co., no. 22950) and once through a 325-mesh screen. The solids content of the coating is determined and then adjusted to 55.0% by adding water.

The coating color was applied to the substrates with a 1.5-mil Bird applicator bar. On aluminum foil the built-in clearance of the Bird bar dictated the coat weight. On MF, because better coat weight control was desired, either the 4 by 5 in. sheets of MF or the zones where the supporting surfaces of the Bird bar traveled were shimmed with thin strips of metal stock.

In preliminary experimental work it became apparent that the coating was considerably stronger in TTS than was the MF for adhesive ratios (parts adhesive/100 parts pigment, by weight) in the range of commercial interest, i.e., above 10. To determine the internal TTS of the coatings on MF it was therefore necessary to remove the MF.

The coatings on MF, after drying at 73°F. (22.8°C.) and 50% R.H. (unless otherwise specified), were isolated from MF by the ethyl acetate treatment developed by Kraske (*1*). After the isolated coating was removed from ethyl acetate and dried, it was stored in humidistats at a known relative humidity, 52% R.H. in most cases. Kraske (*1*) concluded that the ethyl acetate isolation method "accomplishes essentially complete solution of the rawstock without detectable effects upon the strength, the clay particle orientation, the surface area, and the pore distribution of coatings."

Determination of Transverse Tensile Strength

The determination of the internal TTS of clay-starch coatings required the development of a new method based on an old principle. The principle is that TTS can be measured by cementing flat, solid surfaces to either side of the specimen and pulling the specimen apart in a tensile tester. Of the many versions of this test in existence none was satisfactory for testing the internal TTS of clay-starch coatings of commercial

thickness. The difficulties, which were overcome by the newly developed method, were mainly (1) undesirable adhesive properties, (2) lack of means for reproducible assembly of the test cylinders or blocks and the specimen, and (3) lack of means of applying a uniform, simple transverse tensile stress to the specimen.

The adhesive difficulties were solved by a relatively

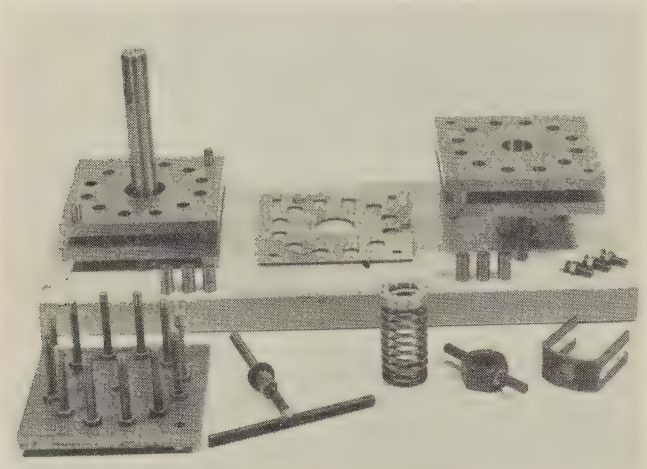


Fig. 2. Apparatus for mounting samples for the evaluation of transverse tensile strength: general layout of components

new adhesive of the epoxy type, Armstrong A-1, made by Armstrong Products Co. The other difficulties were overcome by designing and building (1) an apparatus for mounting samples shown in Figs. 2 to 5 and (2) alignment devices for use in a tensile tester, Fig. 6. By means of the mounting apparatus, 12 specimens at a time can be cemented to steel test cylinders $^1/_2$ in. in diameter by 1 in. long.

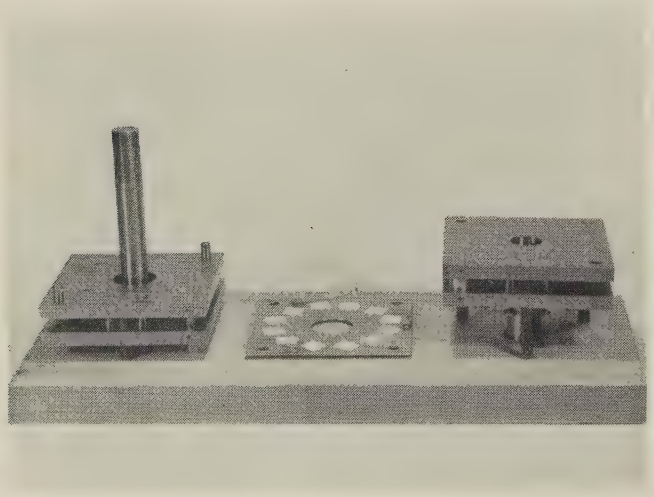


Fig. 3. Apparatus for mounting samples for the evaluation of transverse tensile strength: ready for the application of adhesive to test cylinders

To summarize the test, the general procedure is to:

1. Prepare the specimens of coating.
2. Stir the activator into the epoxy resin and apply the adhesive to the test cylinders positioned in "the apparatus for mounting samples."
3. When the adhesive has cured to the desired degree, assemble the apparatus and place it in a vacuum desiccator.
4. Evacuate the desiccator and clamp the specimens.
5. Remove the assembled apparatus from the desiccator and cure the adhesive in an oven for 2 hr. at 74°C.
6. Remove the test cylinders from the apparatus and carefully trim the edges of the specimens and the adhesive flush with the walls of the test cylinders.
7. Attach the mounted specimens to the aligning devices in the tensile tester (Baldwin-Southwark) and test at a load rate of 500 lb. per min.
8. Examine the fractures with a dissecting microscope to estimate visually the location of failure.
9. Quantitatively remove the samples of coating (with a camel's hair brush and water) from the test cylinders to determine the extent of adhesive penetration and the location of failure.

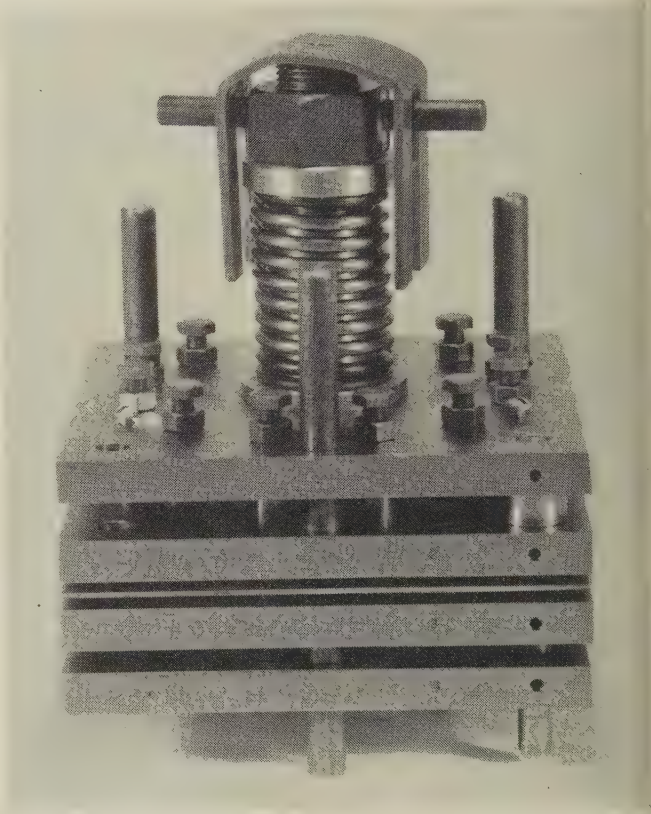


Fig. 4. Apparatus for mounting samples for the evaluation of transverse tensile strength: fully assembled

The TTS of the isolated coating was found to be relatively independent of (1) the relative humidity at which the specimens were conditioned, (2) the absolute pressure at clamping, and (3) the rate of loading during testing. Because precision was improved, the specimens were mounted in a partial vacuum of 8 cm. mercury absolute pressure.

Adhesive penetration in the TTS test is controlled by the clamping pressure and the preliminary cure time before the specimens are bonded. Two conditions of penetration were used: (1) negligible penetration—4 lb. per sq. in. and 1 hr. 46 min. preliminary cure, and (2) slight penetration—12 lb. per. sq. in. and 1 hr. 2 min. preliminary cure. The TTS data obtained in this

way were used to construct strength profiles of the coatings.

Net Adhesive Ratio

The net adhesive ratio, which is defined as the adhesive ratio of the coating layer after drying, was found by weighing a 20 to 30 mg. sample of isolated coating to the nearest 0.1 mg., extracting the starch, and determining the extracted starch colorimetrically with iodine. The clay was calculated by difference, after correction for volatiles in the coating.

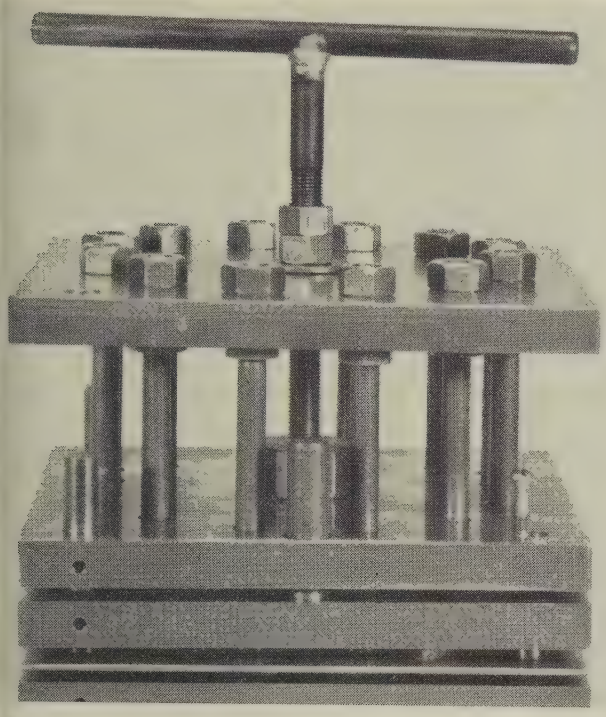


Fig. 5. Apparatus for mounting samples for the evaluation of transverse tensile strength: removal of test cylinders

This colorimetric method for starch is a modification of one described by Browning, Bublitz and Baker (*4*) for starch in paper. The weighed sample of coating is placed in a 50-ml. beaker. A few drops of distilled water are added and the coating macerated thoroughly with a rubber-tipped stirring rod. The stirring rod is rinsed off into the beaker. Distilled water is added to make a total volume of about 10 ml., and the beaker is placed on a steam bath and heated for 30 min. When the beaker has cooled, 1 ml. concd. HCl and enough distilled water to make 25 ml. total volume are added. The beaker is allowed to stand for 1 hr. while the clay settles. The contents of the beaker are then transferred to a fine porosity fritted glass crucible.

The coating residue in the crucible is washed successively with (1) a few milliliters hot, distilled water, (2) 2 ml. 21% HCl, (3) 2 ml. 21% HCl, (4) 1 ml. concd. HCl, diluted with 10 ml. distilled water after 1 min., and finally (5) a few milliliters of hot, distilled water again. The filtrate is transferred quantitatively from the filter flask to a 100-ml. volumetric flask. Five milliliters of concd. HCl and 2 ml. of iodine reagent are added to the flask, and then distilled water up to the mark. The contents are mixed by vigorously inverting the flask ten times. After at least 15 min. but not longer than 1 hr. the optical density of the solution is determined at 580 mμ wavelength. A Bausch and Lomb Spectronic "20" colorimeter was used.

The iodine reagent consists of 7.5 g. KI and 5 g. I_2, diluted to exactly 500 ml. with distilled water.

A calibration curve for Stayco M starch is given in Fig. 7.

The accuracy of the starch determination was about 2%. The initial weight of the coating sample was determined to about 1%. The solids content of the various coatings in this study varied from 98.4 to 99.5%. An average solids content of 99.0% was assumed in calculating the clay by difference. The over-all accuracy of the adhesive ratio determinations was about 2%, whereas the precision for four replicates was about 1%.

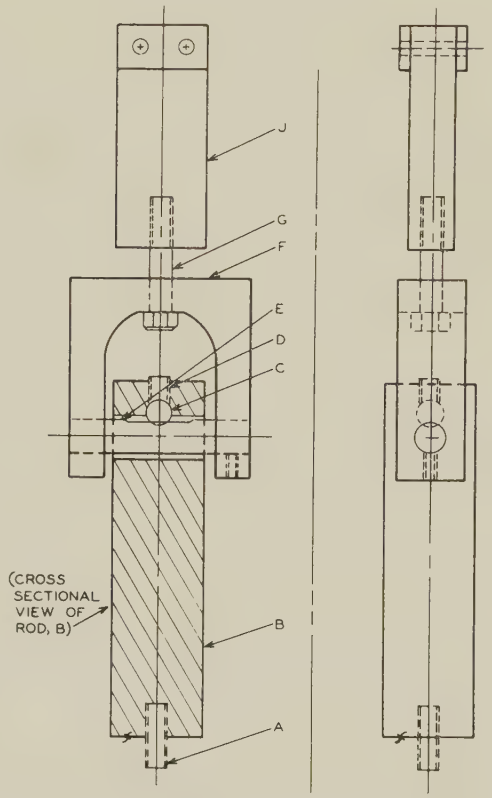


Fig. 6. Device for aligning test cylinders during transverse tensile test, (two required)

EXPERIMENTAL RESULTS AND DISCUSSION: THE EFFECTS OF CERTAIN VARIABLES ON COATING TTS

TTS Versus Pigment Particle Size and Substrate Pore Size

Coatings prepared with the three clay fractions were applied to the three types of MF and to aluminum foil. The initial adhesive ratio was 12, and the coatings were dried by natural convection at 73°F. (22.8°C.) Coat

weight was varied from 10 to 30 lb. per ream (25 × 38—500), but most data were obtained at an average coat weight of about 20 lb. per ream.

Pigment Particle Size

Hemstock and Swanson (*5*) have demonstrated that pick strength is unrelated to pigment surface area or particle size for clay-starch coatings on aluminum foil.

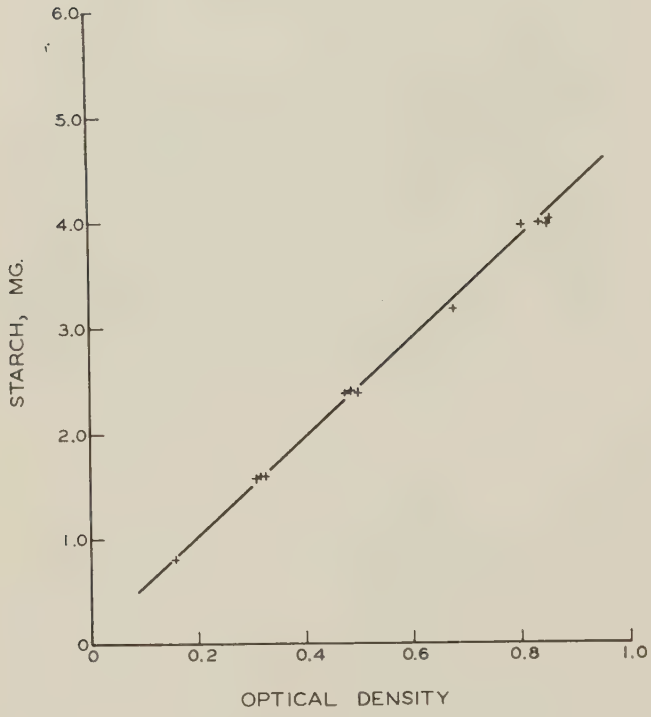


Fig. 7. Calibration curve for Stayco M starch (Colorimetric method using iodine)

The results of the coatings on foil summarized in Table II indicate that TTS also is independent of pigment particle size for coatings on nonporous substrates.

The low TTS values for 12MF* may be due to (1) adhesive migration and/or (2) particle alignment. Because coating 12MF was inadvertently dried at a much lower relative humidity† than the other coatings, the driving force for evaporation of water at the surface of the coating was greater. As Dappen (*6*) has shown, increased rates of drying tend to cause adhesive to migrate toward the surface zones of evaporation. For coatings on foil, migration due to drying can proceed only away from the foil-coating interface and toward the air surface. It is significant that thinner coating samples failed randomly within the coating and at higher strength, whereas thicker samples failed near the foil at lower strengths. The layer weakened most by adhesive depletion due to migration would be expected to occur at the foil-coating interface. Also, adhesive migration effects would be expected to be more pronounced in thicker coatings.

Greater particle alignment may be associated with

Table II. TTS Versus Pigment Particle Size (Coatings on Aluminum Foil)

Coating	*Pigment particle size, μ*	*Average coat weight, lb./ream*	*TTS, lb./in.²*	*Location of failure in coating*
12SF	0.0–0.5	28	2060 ± 140	Random
12MF[a]	0.5–1.0	16[b]	1400 ± 400[b]	Near foil on thick ctgs., random on thin ctgs.
12LF	1.0–2.0	27	2070 ± 90	Random

[a] This coating was dried at less than 10% R.H., whereas the other two coatings were dried at 50% R.H.
[b] There were large variations in coat weight among the samples in this set. Thin coatings gave high TTS values, thick coatings gave low TTS values.

greater adhesive depletion by a closer packing of the platelets. The ability of the clay particles to assume a close-packed arrangement may be greater for the 0.5 to 1.0 μ fraction than for the other two fractions. Kraske (*1*) found that the largest clay fraction had poorer particle orientation than the other two fractions for coatings on MF, type HA. The data were not conclusive on the two smaller clay fractions but seemed to indicate that the medium fraction, 0.5 to 1.0 μ had the greatest degree of orientation of the three fractions.

If increased particle alignment can result in a zone of weakness, it suggests that present concepts of coating strength may need revision. For example, it has been suggested by Hemstock and Swanson (*5*) that very perfect particle alignment is responsible for strong void-free coating adjacent to the foil surface. The implication seems to be that clay and starch make equal contributions (on a volume basis) to strength so that denser coatings are inherently stronger. The results presented above indicate that the volumetric distribution and configurations of the starch are of primary importance and that replacement of starch by clay, volume for volume, weakens the coating.

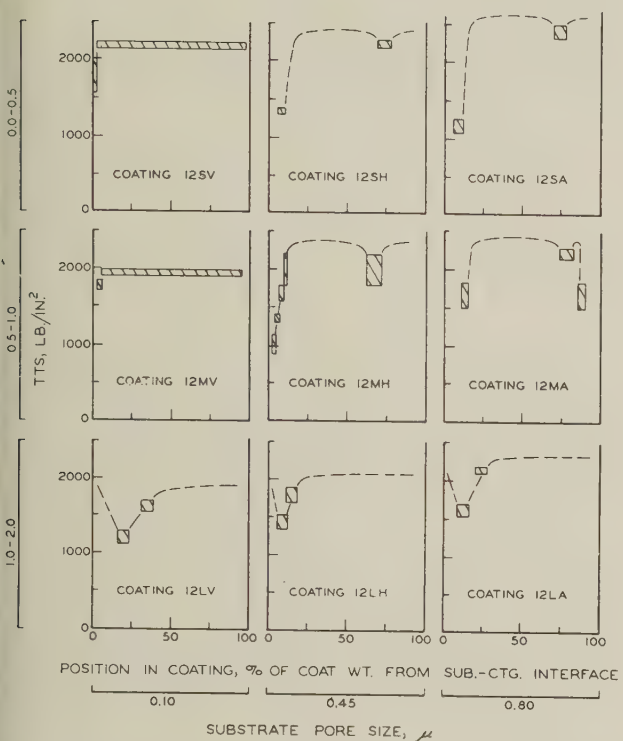
If particle orientation induced by the substrate were the primary cause of the weakness in coating 12MF, it would seem reasonable to expect that TTS would be independent of coat weight or thickness. But TTS varies markedly with coating thickness: therefore, adhesive migration due to drying forces is the more plausible explanation; particle orientation, if a factor, would be the result of the adhesive redistribution rather than the cause.

The results for the coatings on porous substrates is shown graphically in Fig. 8. By the nature of the TTS test it is impossible to measure the strength at the actual surfaces of the coatings, although the surface strength may be approached very closely. Because of this inherent limitation, the TTS profiles of the coatings are drawn only for that portion of the coating not impregnated with the epoxy adhesive during the TTS test. The TTS of the layers of the coating where failure did not occur can be estimated indirectly as greater than that of the zone of failure. The dashed line is given to indicate a *minimum* TTS for that loca-

* Meaning of coating symbols, e.g., 12MF—the two digits represent the initial adhesive ratio of the coating; the first letter stands for the clay fraction used in the coating; the second letter identifies the substrate to which the coating was applied (F = aluminum foil; V, H, and A are types of MF); and the subscript HAB indicates that the coating was dried with a hot air blast.
† Because of equipment failure in the constant humidity room.

ion in the coating; the actual TTS may be much reater.

The cross-hatched rectangle shows two things about he coating: (1) Where failure occurred is indicated ith an accuracy of about 2 lb. per ream by the hori- ontal position and width of the rectangle. (2) The TTS



ig. 8 TTS versus pigment particle size and substrate pore size

; shown by the vertical position of the rectangle, the eight of the rectangle being the 95% confidence limits f the mean. If the behavior of the central 90% of the oating is taken as a criterion, there seems to be a def- nite relationship between TTS and pigment particle ize, as shown by coatings 12SV, 12MV, and 12LV; the TS increased as the particle size decreased. The layers f coating adjacent to the substrate in 12SV and 12MV ave about the same TTS; this suggests that the trength of these layers is dictated primarily by the sub- trate and its effect on vehicle penetration.

Coatings with the largest clay fraction behaved in a istinctly different manner than did the coatings hav- ng the smaller clay fractions. Failure occurred in a ell-defined zone within the coating. The location of he zone of failure varied with the substrate pore size nd with the testing conditions, due perhaps to rein- orcement of the coating by penetration of the epoxy ad- esive, but was generally about $^1/_{10}$ to $^1/_3$ the coat eight from the substrate-coating interface.

The explanation that seems most tenable is that in ne direction starch has left the zone of weakness due o vehicle penetration of the substrate and in the other direction starch has migrated with the vehicle toward the air surface and/or become immobilized before it could be drawn toward the substrate. The resulting zone of depleted starch content would be expected to have less TTS than adjacent zones having more starch. There is a striking connection between this behavior and Kraske's (*1*) pore structure analysis for this large particle size coating. He visualized this coating having constricted, or ink-bottle shaped, pores in order to explain his pore volume and internal surface area data. He pictured constricted pores with openings of two orders of magnitude in size but had no evidence to suggest a preferential location of either size opening. The zone of weakness revealed by TTS testing would be the logical location of large pores, with large openings probably on the substrate side. The smaller constricted pores could be the interparticle voids opening to the air surface of the coating.

The greater strengths of the smaller clay fractions is explainable on the basis of adhesive retention and distribution. On a volume basis, the starch concentration at the zones of failure in 12LV may be less than the starch concentration in 12MV because of the greater void volume found by Kraske (*1*) in the coating of the largest clay fraction. The importance of *volumetric* starch concentration is again suggested.

Substrate Pore Size (Large Clay Fraction)

It seems appropriate to rank coatings 12LV, 12LH, and 12LA with regard to TTS by the strengths of the weakest zones. Clearly, the TTS of a clay-starch coating increases with increasing substrate pore size, under the conditions of this study. At first sight this result seems contrary to the expected behavior. The explanation suggested is as follows.

For a given coating and substrate material the velocity of vehicle penetration, as derived by Washburn (*7*), is directly proportional to the square root of the pore diameter after a given time of penetration. Therefore, for substrates with similar void fractions but different pore sizes, the volume rate of penetration per unit area of surface coated should vary directly with the square root of the pore size.

Thus, near the instant of application the vehicle of coating 12LA penetrates about $^1/_3$ more rapidly than 12LH and nearly three times faster than 12LV. The effects of this difference in rate of penetration seem to be reflected in the structure of the coating. The pigment particles in 12LA were immobilized in a few seconds; visually, the coating was observed to have "set" i.e., lost its wet look. Coating 12LV required a couple of minutes to lose its wet look. The rate at which pigment immobility is developed seems to affect the density of the coating and the amount of starch retained with the clay.

At a low rate of penetration, the vehicle is visualized as being drawn from the coating layer directly adjacent to the substrate. When this layer has compacted to a rigid structure, vehicle is supplied from a layer farther from the substrate, and so on, until the coating as a whole has set. Adhesive penetration and coating compaction are maximized.

At a high rate of penetration, the vehicle may be withdrawn from the coating so rapidly that the layer

adjacent to the substrate can fill the requirement only instantaneously; and the vehicle is almost immediately supplied from the total coating thickness. As vehicle leaves the coating, the pigment particles, and any layers of adhesive immobilized adjacent to the particle surfaces, coalesce to form a rigid structure. The vehicle flowing between and around fixed or slowly moving clay particles will assume a velocity distribution such that the greatest rate of flow will occur in the center of the channels available for flow. Saturated flow could therefore end while a large portion of the vehicle still exists around the pigment particles.

When saturated flows ends and air enters the coating, i.e., the wet look disappears, vehicle must flow around particles rather than between them; and the resistance to flow becomes much greater. It is suggested that the major loss of adhesive to the substrate occurs in the saturated state of flow and that low rates of penetration favor the maintenance of this type of flow, even to the extent that a greater volume of vehicle penetrates a substrate of small pore size than it does a substrate of large pore size.

Cutting 0.5 μ-thick cross sections of coatings such as 12LH and 12LA with a thin-sectioning microtome and examining the extent of starch penetration by staining with iodine revealed an enlightening fact. The adhesive had penetrated farther into the substrate with the larger pores than was the case with the smaller pores. This indicates that depth of adhesive penetration into the substrate is not necessarily proportional to the quantity of adhesive lost from the coating, inasmuch as coatings on smaller pore-size substrates tended to lose more starch. It is not surprising that Casey and Libby (*8*) did not find a correlation between depth of adhesive penetration into the bodystock and wax pick strength for starch-clay coatings on paper.

The thinner but more concentrated layer of starch found in smaller pore-size substrates may contribute to pick strength by strengthening the substrate-coating bond and the surface layer of the substrate.

Interaction Effect

In the coatings previously discussed, the vehicle penetrated the substrate but the pigment was left behind. When the pigment also penetrates the substrate, the coating strength and structures are altered markedly. This is evident for the smaller clay fractions on the larger pore-size substrates (see coatings 12SH, 12SA, 12MH, and 12MA). Minimum TTS values for 12SH, 12SA, 12MH, and 12MA are not too reliable because the roughness of the substrate side of the coating (due to pigment penetration) may have prevented the epoxy adhesive from making intimate contact with the complete coating surface.

Table III. Apparent Density Data, g. per cc.

Coatings on MF, initial adhesive ratio = 12, dried by natural convection at 73°F.

Pigment particle size μ, e.s.d.	*Substrate pore size, μ* 0.10	0.45	0.80
0.0–0.5	1.54[a]	1.48[a]	1.12[a]
0.5–1.0	..	1.60	1.18
1.0–2.0	..	1.35[a]	1.29[a]

[a] Data provided by D. J. Kraske (*9*).

The most noticeable difference in these coatings, compared to the coatings without pigment penetration, is the zone of weakness located in the upper third of the coating, i.e., toward the air surface of the original coating. This new zone of weakness follows the same trends of TTS versus substrate pore size and pigment particle size as were previously described. TTS is higher for the smaller clay fraction and for the larger pore-size substrate.

Table IV. Net Adhesive Ratio Versus Pigment Particle Size and Substrate Pore Size

Coatings on MF, initial adhesive ratio = 12, dried by natural convection at 73°F.

Pigment particle size, μ, e.s.d.	*Substrate pore size, μ* 0.10	0.45	0.80
0.0–0.5	9.8	11.0	11.3
0.5–1.0	9.5	10.7	11.2
1.0–2.0	9.8	11.4	11.4

These coatings set quite rapidly and appear to have a more open structure. When the pigment particles are much smaller than the pores, as in coatings 12SA and 12MA, the apparent density as determined by the mercury intrusion method developed by Kraske (*1*) falls off sharply. Tentative apparent density data available for these coatings are summarized in Table III. For any given clay fraction there seems to be a trend toward lower densities as substrate pore size increases. The low apparent densities for the large clay fraction are probably due primarily to poor particle packing rather than pigment penetration into the substrate.

Table IV summarizes the net adhesive ratio for the nine coatings.

Substrate pore size seems to be an important factor in adhesive penetration. The adhesive lost by substrate penetration is nearly the same for all three coatings on a given pore-size substrate. The 0.5 to 1.0 μ clay fraction lost more starch than either of the other two fractions for a given substrate. This may be related to the increased tendency to assume a preferred orientation reported by Kraske (*1*) for this fraction. It will be recalled that this clay fraction had less TTS than the other fractions in coatings on foil.

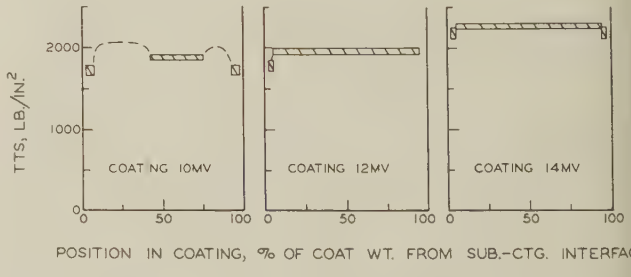


Fig. 9. TTS versus initial adhesive ratio

Initial Adhesive Ratio

Coatings with initial adhesive ratios of 10 and 14 were applied to MF, type VC, and to aluminum foil. The 0.5 to 1.0 μ clay fraction was used, and the coatings were

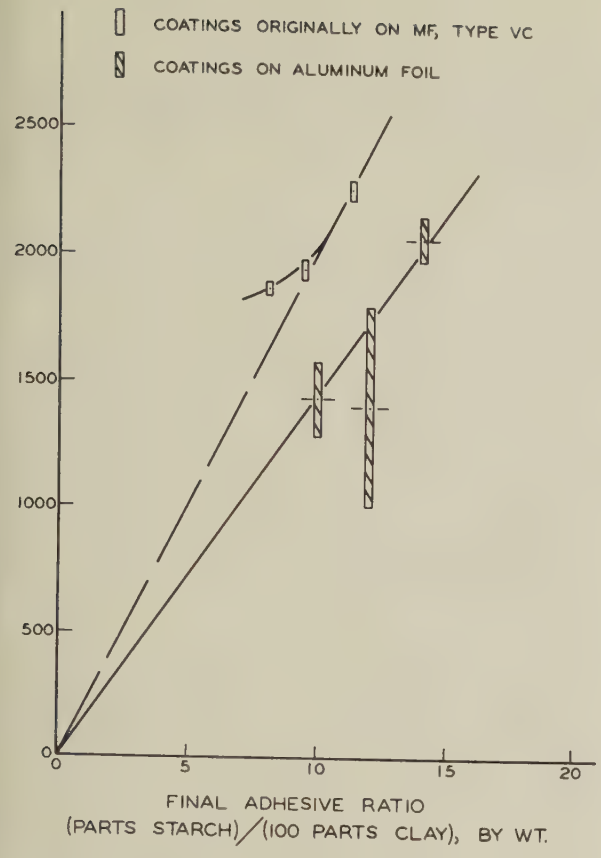


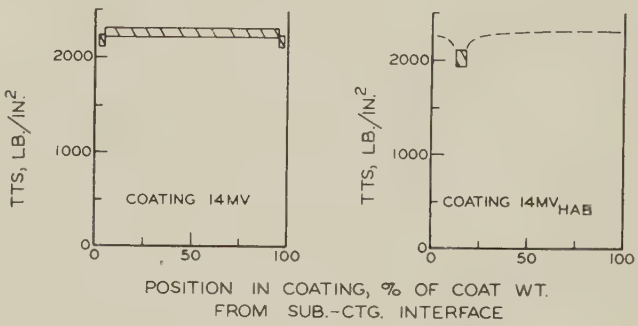
Fig. 10. TTS versus final adhesive ratio

ried by natural convection at 73°F. These two coatings are compared in Figs. 9 and 10 with a similar coating, previously described, having an initial adhesive atio of 12.

The most obvious feature of these coatings is that TS increased with increasing initial adhesive ratio, or ith increasing *net* adhesive ratio. The data shown in ig. 10 suggest that the relationship between TTS and et adhesive ratio may not be linear for coatings on MF. he low significance level of the TTS data for coating 2MF enables one to draw a wide variety of curves to t within the 95% confidence limits. The low relative umidity at which 12MF was dried has been mentioned bove as a possible cause of the low TTS of 12MF. It interesting and perhaps significant that a straight line an be drawn through the origin and the data points for oatings 10MF and 14MF. TTS may be directly proortional to the final adhesive ratio for clay-starch coatngs on foil when the adhesive distribution is uniform.

The second feature shown in Fig. 10 is that for a given dhesive ratio the coatings on MF have greater TTS an the coatings on foil. This effect is contrary to the ndings of Hemstock and Swanson (*5*) for pick strength. ick strength as measured with the IPC bonding rength tester, and probably other press simulation tests, is sensitive to the physical properties of the bodystock. This was shown by the following demonstration:

A clay-starch coating (initial adhesive ratio = 12, 0.5 to 1.0 μ clay fraction, dried in air at 73°F.) was applied to MF, type HA, and to aluminum foil. Strips of these two coatings, still on their original substrates, were tested with the IPC bonding strength tester using the prefilming technique described by Wink, Shillcox, and Van Eperen (*10*). The coating on MF gave a viscosity-velocity product (VVP) of less than 10, and the coating on aluminum foil gave a VVP greater than 60. However, when the coating originally on MF was mounted coating side down on aluminum foil with a thin layer of Trim Adhesive (Minnesota Mining & Mfg. Co.)



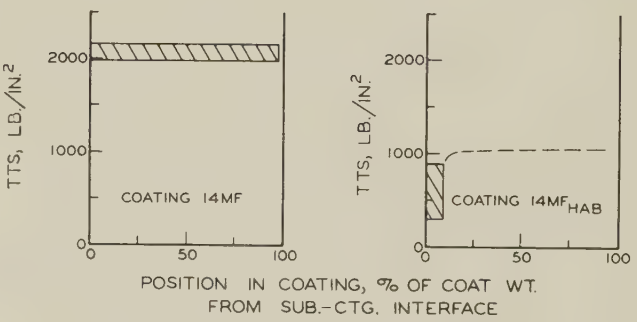


Fig. 11. TTS versus method of drying

and the MF removed with ethyl acetate, the VVP obtained was greater than 60.

Further consideration of the influence of substrate properties on the pick strength of coated paper is needed. Mere retention of adhesive within the coating or even preferential particle alignment do not insure high TTS, and their influence on pick strength certainly needs closer scrutiny.

Method of Drying

Drawdowns of coating 14MV were dried by a blast

Table V. TTS Versus Method of Drying

Coating	*Method of drying*	*Substrate pore size, μ*	*Net adhesive ratio*	*Coat weight, lb./ream*	*Coat weight penetrated, lb./ream*	*TTS, lb./in.²*	*Location of failure in coating, % of cost wt. from sub. ctg. interface*
14MV	Natural convection at 73°F.	0.10	11.3	23 ± 3	0.8	2170 ± 60	(near surfaces)
				23 ± 3	2.1	2260 ± 40	(random)
14MV$_{HAB}$	Hot air blast at 200°F.	0.10	12.4	22 ± 3	0.3	2020 ± 80	17
				23 ± 6	0.7	2030 ± 60	14
14MF	Natural convection at 73°F.	(Foil)	14.0	24	..	2060 ± 90	(random)
14MF$_{HAB}$	Hot air blast at 200°F.	(Foil)	14.0	31	..	600 ± 300	(narrow zone adjacent to foil)

of 200°F. (93.4°C.) air from a home-type hair drier, as well as by natural convection at 73°F. Hot-air-blast drying required 1 min. for coatings on MF and two minutes for coatings on aluminum foil. The results are summarized in Table V and Fig. 11.

The increased rate of drying reduced the TTS of both the coating on MF and the one on aluminum foil. The coating on foil lost a much larger portion of its TTS than did the coating on MF. Apparently, the porous substrate competes with the capillary forces generated by the drying action. Note that the blast-dried coating on MF, $14MV_{HAB}$, retained significantly more of its original adhesive than the coating on MF dried more slowly. The layers near the surface of the coating on MF appeared to be strengthened by blast drying, indicating that part or all of the increased starch retention is in the surface layers.

The TTS distributions of these coatings correlate very closely with the known or postulated adhesive distributions for similar coatings.

The physical structure and properties of coatings 14-MV and $14MV_{HAB}$ are different. Qualitative evidence of this is that the blast-dried coating is much more brittle. Very little bending is needed to break the isolated coating. Increased starch concentrations at the two surfaces of the coating could explain such brittleness, since thick films of Stayco M starch are so brittle they shatter like glass. The thinner the film of Stayco M the more flexibility and toughness it seems to have. Additional evidence is provided by the depth of epoxy adhesive penetration. The much lower penetration into $14MV_{HAB}$ suggests that the surface pores are more constricted, probably by starch.

Examination of stained cross sections showed that the depth of starch penetration into the MF was less for blast-dried coatings than for coatings dried in air at 73°F. This agrees with the net adhesive ratio data.

Apparent density determinations by the mercury intrusion method indicated that the blast-dried coating had about the same void volume as the one dried at 73°F. The slightly lower apparent density of the blast-dried coating was accounted for by the increased starch content.

Coat Weight

Careful examination of the TTS data for coatings on MF revealed no significant effects due to variations in coat weight from 10 to 30 lb. per ream. Kraske (*1*), in his study of coating structure, found coatings of 10, 20, and 30 lb. per ream to be nearly identical.

The TTS data for coating 12MF, on aluminum foil, showed a strong dependence on coat weight; the thin coatings gave much higher strengths than did the thicker ones. Dappen (*6*) has shown that more starch migrates away from the layer of coating adjacent to the substrate in thicker coatings than in thin ones.

This loss in TTS with increasing coat weight might be found in coatings on porous substrates (1) if the coating is dried extremely rapidly and/or (2) if the substrate has very small pores.

CONCLUSIONS

The following conclusions are based on this study of the internal TTS of clay-starch coatings:

1. A method and an apparatus have been developed for measuring the internal TTS of clay-starch coatings.

2. The TTS of the clay-starch coatings isolated from MF with ethyl acetate is relatively insensitive to conditioning at different relative humidities and to the rate of loading used in the testing.

3. Other factors being equal, a coating applied to a substrate with small pores loses more adhesive to the substrate and has less TTS than does the same coating applied to a substrate with larger pores.

4. Other factors being equal, coatings made with larger clay particles and applied to a porous substrate have less TTS than coatings with smaller clay particles.

5. On a nonporous substrate the TTS of a coating is not directly related to pigment particle size.

6. The TTS of clay-starch coatings increases with increasing adhesive concentration.

7. When applied to a porous substrate, coatings dried with a hot-air blast retain more adhesive but have less TTS than coatings dried by natural convection at room temperature.

8. When applied to a nonporous substrate, coatings dried with a hot-air blast have less TTS and fail nearer the substrate than coatings dried by natural convection at room temperature.

9. Variations in coat weight have little effect on the TTS of clay-starch coatings on porous substrates such as MF.

10. On nonporous substrates thick coatings tend to be weaker in TTS than thin coatings, and the failure occurs near the substrate instead of randomly.

11. The amount of starch lost from a coating is not necessarily related to the depth of penetration of starch into the substrate.

12. Greater particle orientation does not necessarily increase TTS.

13. The TTS of clay-starch coatings seems to be largely governed by the final adhesive distribution in the coating. The distribution of voids and the degree of particle orientation apparently affect TTS through their effects on the adhesive distribution.

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Preparation of Dissolving Pulp from Sugarcane Bagasse

ANDRE H. LOCUS

High-grade experimental pulps for production of viscose rayon were prepared from bagasse by the prehydrolysis-sulphate process. A study of hydrolyses by sulphuric acid, nitric acid, water, and steam is reported. Results emphasize the importance of hydrolytic conditions upon effectiveness of pentosan removal. High-grade dissolving pulps with 91 Hunter brightness and containing 95% alpha-cellulose, 4% pentosans, and 0.05% ash were obtained. Yield from depithed bagasse was about 33%, comparable to that from wood. The bagasse pulps were spun into rayon and the yarns evaluated. The pulps had characteristics needed for the manufacture of viscose rayon.

For many years cotton linters were used as the only raw material for the manufacture of viscose rayon. It was generally believed that replacement of cotton by wood would not be economically feasible or technically possible. Today, wood pulps provide the major amount of raw materials for the rayon industry around the world. First, softwood pulped by the sulphite process replaced cotton. Then, since World War II, the greater demand for rayon and the development of new techniques have made possible the commercial production of bleached hardwood pulps for manufacture of rayon. At present high-grade dissolving pulps made from hardwood by the prehydrolysis-sulphate process have gained wide acceptance throughout the industry.

New sources of raw material for rayon are desirable, however, and for many foreign countries sugarcane bagasse has the economic advantage among agricultural residues of being already collected at the sugar mills.

The chemical composition of depithed bagasse is somewhat similar to hardwood. The main difference from softwood is in its higher pentosan content. The need of removing these additional pentosans accounts for one of the major difficulties in making a good grade dissolving pulp. The pulping process used in these studies is an adaptation of the prehydrolysis-sulphate process used for hardwoods. In view of the proved success of this process for production of dissolving pulps from hardwoods, it was chosen as the most logical one for the preparation of high alpha pulps from agricultural residues. This process with appropriate modification of the operating variables permits the preparation of bagasse pulp of quality comparable to that of high-grade dissolving pulp from wood. The conditions of this process, such as temperature, time of reaction, and chemicals used, are practical and present no unusual engineering problems.

Prior to chemical treatment, bagasse must be depithed if a high-quality pulp is to be obtained. Judging from chemical composition alone, pith should make a pulp as good as the fiber; cellulose, lignin, and pentosan content do not differ greatly for whole bagasse, depithed fiber, and the pith alone. However, the thin-walled pith cells, because of their physical structure, are fragile and tend to break easily during processing. These cells and cell fragments are highly absorbent so that they retain dirt, as well as pulping chemicals. Once contaminated in such a manner, it is almost impossible to purify the pulps by washing and filtering because of unfavorable drainage characteristics. Cellulose sheets containing pith are hard and horny and they do not steep uniformly; their physical strength when wetted in caustic soda is so low that pressing of the alkali-cellulose during the conversion to rayon is very difficult.

Depithing methods need not be discussed here. The Northern Utilization Research and Development Division, Peoria, Ill., has made a very thorough study of this subject and has developed several practical and economical methods for effecting the needed separation (*1*). The preferred method makes use of a disk mill to loosen the pith from the fiber; the actual separation, wet or dry, is done by screening. The pith by-product of the operation can be recovered and may be used as a fuel or mixed with molasses and other additivies, pelleted, and sold as a feed (*2*).

MATERIAL AND METHODS

The raw materials used in these studies were depithed bagasse from Florida and the Philippines. About one half of the work was done with fiber from

Table I. Physical Composition of Whole Bagasse

	Florida, %	*Philippine*, %
Fiber	65	67.5
Pith	25	27.5
Dirt and water solubles	10	5.0

Florida bagasse depithed by the Peoria hydrapulper process (*1*). The remainder of the work was done with fiber from Philippine bagasse depithed by the disk mill process. Although depithed by different procedures, the separated fiber from the two sources exhibited no significant differences in composition or performance. The Philippine material contained about 3% more lignin than the Florida bagasse and thus required slightly more chemical during cooking to duplicate the results obtained with the Florida fiber. Proportions of physical components of the original bagasse are presented in Table I. Proximate chemical analyses of Florida and Philippine bagasse, as well as the depithed fiber and pith, are presented in Table II.

Except when otherwise specified, hydrolyses with acids were carried out on 200-g. samples, in 2-l. stainless steel digesters heated in an oil bath. For experiments with sulphuric acid, a glass container was put

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inside the digester. At the end of the hydrolysis period, the digesters were cooled immediately by immersion in a tank of water. The materials were washed several times with distilled water, then with water made slightly alkaline with a few drops of ammonia, and again with distilled water. The hydrolyzed materials were air dried and moisture determinations were made on the air-dry materials for calculation of yields.

Hydrolyses with water and steam were carried out in a 1.6-cu. ft. rotating stainless steel digester equipped for direct and indirect heating with steam. Each sample was about 7.5 lb. of air-dry bagasse fiber. At the end of a hydrolysis period the digester pressure was immediately released and the contents dumped and washed three or four times with cold tap water in a wooden tank equipped with a perforated bottom.

Each batch of washed hydrolyzed fiber was allowed to drain for a few hours, mixed thoroughly, weighed, and sampled in triplicate for moisture content. Monoethanolamine cellulose (MEA) was determined on the hydrolyzed fiber according to the method of Reid, Nelson, and Aronovsky (*3*) and the alpha-cellulose content determined on the MEA cellulose according to TAPPI Standard T 420 m-48 (*4*). Pentosans were determined according to TAPPI Standard T 223 m-48 and calculated by the formula $[7.5 \times n\ (v_2 - v_1)]/w$ in which the symbols have the same meaning as those in the TAPPI method. It should be noted that no correction is made for any hydroxymethyl furfural that might be formed from cellulose. The pentosan values reported therefore might be higher than actual so that the true effectiveness of the hydrolysis treatment may be greater than indicated.

Table II. Proximate Chemical Composition of Florida and Philippine Bagasse

	Florida			*Philippine*		
	Whole, %	*Fiber, %*	*Pith, %*	*Whole, %*	*Fiber, %*	*Pith, %*
Cellulose (MEA)	50.0	60.0	52.0	55.0	61.0	55.0
Alpha-cellulose	40.0	45.5	40.5	40.5	44.5	41.0
Pentosans in alpha-cellulose	8.0	9.0	8.5	7.5	9.5	8.0
Alpha-cellulose corrected for pentosans	36.8	41.4	37.1	37.5	40.3	37.7
Lignin	19.0	19.0	19.0	22.5	21.8	22.5
Pentosans	28.0	28.5	28.0	28.0	28.5	28.0
Ash	2.2	2.0	3.0	3.0	2.0	5.5
Alcohol-benzene (1:2) solubles	7.0	2.6	3.0	3.0	2.0	2.5

Alcohol-benzene solubility was run according to TAPPI Standard T 6 m. Lignin was determined on material according to the procedure described by Aronovsky, Nelson, and Lathrop (*5*). Viscosity and degree of polymerization characteristics were determined on the pulp according to the nitration method of Alexander and Mitchell (*6*).

After an investigation of various hydrolyzing conditions including a number of hydrolyzing agents, the more promising conditions were used in pilot plant operations to study pulping and bleaching conditions. Since 15 lb. of separated fiber were required to provide a 5-lb. yield of purified pulp, two 7.5-lb. batches of fiber were hydrolyzed separately in the 1.6-cu. ft. stainless steel digesters. The two batches of hydrolyzed fiber were combined for a single pulping run with 18% kraft chemicals based on depithed fiber. Brown stock from digestions was washed in a wooden tank equipped with a perforated bottom and then screened on a Valley laboratory pulp screen, 0.008-in. cut. Chlorination of screened pulp was carried out in a 50-gal. rubber-lined tank and the other bleaching stages in a 50-gal. glass-lined kettle. The bleached pulp was finally sheeted in a Valley handsheet mold (6 by 9 in.) and dried on a steam-heated drum. Final bleached pulps from the pilot plant operations were converted to viscose rayon, spun to yarn, and evaluated by the Von Kohorn International Corp.

PENTOSAN HYDROLYSIS

Hydrolysis is the most important step in the preparation of alpha-pulp from bagasse because hydrolysis conditions really determine the quality characteristics of the final pulp. Two different things happen during this operation: First, the pentosans are degraded, about 75% being solubilized and the remaining pentosans being attacked to such an extent that most are soluble in the alkaline liquor during the cooking process. Second, besides the desirable action, there is degradation of the cellulose that cannot be avoided completely and depends upon the hydrolyzing agent and the conditions of hydrolysis. Alpha-cellulose is degraded into products such as beta-cellulose which can never be completely eliminated by simple methods of purification. In other words, drastic conditions for hydrolyses contribute to low pulp yields and inferior pulp quality. Such pulps are deficient in alpha-cellulose and have significant contents of beta-cellulose. Too drastic conditions for hydrolyses result also in brittleness of fiber which in turn produces considerable fine material and causes trouble during subsequent purification of the pulp.

Effects on the bagasse fiber of hydrolyses by sulphuric acid, nitric acid, water, and steam under different combinations of temperature, time, and acid concentration are shown in Table III. Data on yields of hydrolyzed fiber, pentosans and their removal, alpha-cellulose content of the hydrolyzed fiber, and the extent of degradation of the alpha-cellulose are presented. Effective reduction of pentosan content was achieved for all hydrolyzing agents. Pentosan content decreased as the chemical concentration, time, or temperature in the hydrolysis was increased.

If data for pentosan content and yield of fiber for the various hydrolysis treatments are plotted together, a direct linear relationship appears to exist between these two factors. Thus, for the same yields of hydrolyzed fibers following use of different hydrolyzing agents, the pentosan contents of the fibers will be essentially the same. Except under drastic conditions, most of the alpha-cellulose in the original material will be recovered after hydrolysis. This recovery does not mean that cellulose from different hydrolysis processes is identical; our study showed that cellulose degradation varies within wide limits. Cellulose depolymerization occurs differently according to the hydrolyzing agent used and the hydrolysis conditions.

To illustrate, among the series of pilot plant experiments involving various hydrolyzing agents two pulps were selected having nearly equal alpha-cellulose contents. One pulp was prepared from fiber hydrolyzed

Table III. The Influence of Hydrolyzing Agent, Time, Temperature, and Concentration on the Hydrolysis of Fiber from Philippine Bagasse

Temp., °C.	Time, hr.	Concentration, %	Yield, %	Pentosans content, %	Pentosans removed, %	Alpha-cellulose, %	Alpha-cellulose degraded, %
			Hydrolyses by H_2SO_4				
140	1	0.5	68.2	7.1	82.2	56.9	12.8[a]
140	1	0.75	65.1	2.8	93.5	60.5	11.4
140	1	1.00	63.6	2.5	94.5	58.3	16.7
140	1	2.00	62.1	1.6	96.5	51.5	28.1
140	1/4	1.00	65.9	6.7	84.2	58.2	13.9
140	1/2	1.00	64.6	3.1	92.9	59.8	13.2
125	1	0.5	70.0	10.8	73.0	..	..
160	1	0.5	60.9	1.8	96.1	..	..
			Hydrolyses by HNO_3				
140	1	0.3	69.5	9.8	75.7	58.0	9.4
140	1	0.6	65.0	6.6	84.7	58.2	15.0
140	1	0.9	65.1	5.5	87.2	59.0	13.6
140	3	0.3	67.3	7.3	82.5	57.4	13.2
120	3	0.3	74.2	14.1	62.8	..	..
160	3	0.15	61.1	4.3	90.6	56.3	22.7
			Hydrolyses by Water				
160	2	..	78.7	18.9	48.5	..	..
160	3	..	75.0	15.7	58.0	..	..
160	4	..	71.2	11.2	71.5	..	..
170	2	..	69.3	9.8	75.7	58.6	8.8
180	2	..	66.7	5.4	87.1	56.9	14.8
			Hydrolyses by Steam				
170	1	..	72.9	14.0	63.5	57.7	5.5
170	2	..	70.1	7.4	81.5	60.0	5.5
170	3	..	70.8	5.3	86.6	56.3	10.4
180	2	..	67.8	4.0	90.3	54.0	17.7
190	1 1/2	..	66.3	2.4	93.9	46.2	31.2

[a] Alpha-cellulose degraded was calculated as:

$$100 - \frac{68.2 \times 56.9}{44.5} = 12.8\%$$

where 44.5 is the alpha-cellulose content of the Philippine bagasse fiber (Table II).

with sulphuric acid, 0.25% (basis dry fiber), for 60 min. at 125°C.; the other from fiber hydrolyzed with steam for 15 min. at 190°C. The hydrolyzed fibers were pulped and bleached under exactly the same conditions, the pulping being 1 hr. at 150°C. with 18% kraft chemical and bleaching done in seven stages. Characteristics of the hydrolyzed fibers and of the final bleached pulps are shown in Table IV.

Results of these two pulping experiments show clearly that although the characteristics of the two materials are similar after hydrolysis, their celluloses behave in different ways under identical cooking conditions. The material hydrolyzed with sulphuric acid had its alpha-cellulose degraded in such a way that 31.4% was solubilized on cooking while the material hydrolyzed by steam had only 16.3% solubilized by similar cooking. Repeated results of this kind showed that steam hydrolysis was the best way to eliminate the pentosans with a minimum attack on the cellulose.

Table IV. Characteristics of Hydrolyzed Fiber and Dissolving Pulps from Philippine Bagasse

	Hydrolyzed bagasse fiber		Dissolving pulps	
	Sulphuric, %	Steam, %	Sulphuric, %	Steam, %
Yield[a]	78.0	71.9	30.0	35.5
Alpha-cellulose	58.8	60.1	..	..
Alpha-cellulose corrected for pentosans	49.5	53.8	92.1	95.0
Pentosans	15.6	10.5	6.9	4.1
Ash	..	..	0.06	0.05
Brightness	..	..	91.0	90.5
Degree of polymerization	..	..	750	725
Alpha-cellulose degraded[b]	4.5	5.7	31.4	16.3

[a] Basis dry bagasse fiber.

[b] Basis pentosan-free alpha-cellulose in bagasse fiber; e.g., $100 - \frac{92.1 \times}{40.3}$ = 31.4% degraded.

PULPING OF HYDROLYZED FIBER

Some quality characteristics and yields of unbleached pulps prepared from bagasse were investigated as functions of the digestion temperature, digestion time, and concentration of kraft chemicals. The digestions were carried out in the tumbling stainless-steel digester heated with indirect steam. The amounts of kraft chemicals ($NaOH:Na_2S$ = 2:1) employed were based on the dry weights of the depithed bagasse fiber before hydrolysis. The liquid-solids ratio was 6:1. All experiments were run on batches of hydrolyzed fiber prepared under the same conditions of steaming at 190°C. for 15 min. Results of these studies are presented in Table V.

The permanganate number of the pulps decreases rapidly when the percentage of chemical and the time of reaction increases. If the fiber was hydrolyzed with acid, however, in certain conditions the permanganate number of pulps increased when the time of reaction was increased from 1 to 2 hr. In many cases when hydrolysis conditions were too severe, a very low yield would correspond to high permanganate number and hard-to-bleach pulp while best hydrolysis condi-

Table V. Influence of Digestion Time, Temperature, and Chemical Concentration upon Yields and Quality of Unbleached Pulps Prepared from Steam-hydrolyzed Fiber or Sugarcane Bagasse[a]

Temp., °C.	Time, hr.	Amount of chemical, %[b]	Yields: Basis hydrolyzed fiber, %	Yields: Basis depithed bagasse, %	Residual pentosans, %	$KMnO_4$ number	Spent liquor, pH	D.P.
170	1	15	52.3	36.1	3.7	15.5	10.8	..
	1	18	48.7	33.6	3.8	8.4	11.5	925
	1	22	50.5	34.8	..	7.4	12	..
	1	26	49.7	34.3	3.6	7.0	12	..
	1/2	16	49.4	34.1	..	9.6	11.7	1050
	1/2	22	49.1	33.9	3.9	7.4	11.9	1000
150	2	18	54.9	40.8	..	8.4	12.0	1150
	1	18	57.2	42.5	..	9.8	12.0	1150
	1/2	18	59.5	44.2	3.7	11.0	12.0	1300
140	2	18	50.7	35.1	4.0	10.2	11.9	1150
	1	18	50.9	35.2	..	13.6	12.0	1200
	1/2	16	52.8	36.5	..	16.7	12.0	1200
	1/2	18	51.2	35.4	4.3	14.2	12.1	1250
		20	53.3	36.9		13.6	12.0	1250
		22	54.6	37.8	4.3	13.5	12.0	1250
130	2	18	61.1	43.6	..	14.0	11.8	1200
	1	18	61.4	43.8	..	15.9	11.9	1200
	1/2	16	60.8	43.4	..	19.6	12.0	..
	1/2	18	60.9	43.4	4.3	17.0	12.0	1250
	1/2	20	63.8	45.5	..	16.6	11.9	1200
	1/2	22	64.7	46.2	4.4	15.6	11.9	..

[a] These cooking experiments were made with batches of hydrolyzed bagasse fiber that had been prepared under identical conditions of treatment by steam at 190°C. for 15 min.
[b] Kraft chemical basis weight depithed fiber dry basis.

tions would give good yield, low permanganate number, and easy-to-bleach pulps.

BLEACHING AND PURIFICATION

All pilot plant pulps reported were bleached by a seven-stage process:

1. Chlorination-Cl_2 room temperature for 1 hr., 3% consistency
2. Caustic extraction—2% NaOH, for 1 hr. at 70°C., 5.5% consistency
3. First sodium hypochlorite stage—4 hr. at 40°C., 0.5% available chlorine, 5.5% consistency
4. Caustic extraction—0.5%, 1 hr. at 70°C., 5.5% consistency
5. Second sodium hypochlorite stage—4 hr. at 40°C., 0.3% available chlorine, 5.5% consistency
6. Caustic extraction—0.5%, 1 hr. at 70°C., 5.5% consistency
7. Sodium chlorite—2 hr. at 80°C., 5.5% consistency, 0.2% available chlorine, pH kept at 2.5 with HCl

Characteristics of four experimental dissolving pulps prepared from bagasse when sulphuric acid, water, and steam were the hydrolyzing agents are shown in Table VI. Characteristics of commercial pulps prepared from cotton linters, softwood, and hardwood (beech) are presented for comparative purposes. From these data it is evident that the bagasse alpha-pulp prepared through water or steam hydrolysis and followed by kraft pulping had characteristics very similar to those of commercial products. These experimental bagasse pulps prepared on a small pilot plant scale were subsequently converted to rayon in a commercial mill. Tenacity and elongation characteristics of two of the experimental yarns, as well as a normal commercial product of the mill, are shown in Table VII. Characteristics for the experimental yarns are deemed comparable to those of the commercial product.

Table VI. Some Chemical and Physical Characteristics of Experimental Dissolving Pulps Prepared by Prehydrolysis-Sulphate Treatment of Bagasse and of Some Commercial Dissolving Pulps[a]

Type of dissolving pulp	Hydrolyzing agent	Pulping time, hr.	Yield, %	Alpha-cellulose, %	Beta-cellulose, %	Pentosans, %	Ash, %	Viscosity, cp.	D.P.	SiO_2 p.p.m.	Fe^{+++} p.p.m.
Experimental:											
Philippine bagasse	H_2SO_4	1/2	30.0	92.1	6.2	6.9	0.05	8.3	750	..	..
Philippine bagasse	Water	1/2	31.0	94.0	..	4.5	0.04	10.0	840	..	..
Philippine bagasse	Steam	1/2[b]	30.6	95.8	..	2.5	0.06	7.2	700	97	270
Florida bagasse	Steam	2	34.8	95.7	2.5	3.4	0.05	4.9	525	..	..
Commercial:											
Cotton linters	...	..	..	99.1	0.6	..	0.5	..	..	43	150
Softwood	...	..	..	92.1	4.9	1.9	0.05	..	..	36	101
Hardwood (Beech)	...	..	..	92.6	4.3	3.2	0.09	..	..	38	229

[a] Kraft chemical, 18% basis original dry fiber at 170°C.
[b] Pulping at 150°C.

SUMMARY

Dissolving pulps having a high brightness, less than 4% pentosans, more than 95% alpha-cellulose, and 0.1% or less ash have been obtained by steam hydrolysis of depithed sugarcane bagasse fiber followed by pulping with kraft chemicals and bleaching in seven stages. Yields from depithed bagasse fiber were

Table VII. Tenacity and Elongation of Experimental Rayon Yarns (100 Denier, 40 Filament) Prepared from Alpha-Cellulose Pulps Obtained from Florida Bagasse

No.	Yield, %	Tenacity: Dry, g.	Tenacity: Wet, g.	Elongation: Dry, %	Elongation: Wet, %
175	33.6	2.17	1.01	15	21
176	29.3	2.12	1.01	19	26
Commercial	..	1.90	0.94	17	23

about 33% or comparable to that obtained from wood. Characteristics of experimental bagasse pulps are similar to commercial pulps used for the manufacture of viscose rayon. Good quality dissolving pulps were prepared from sugarcane bagasse.

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Kinetics of the Alkaline Degradation of Hemicellulose at Pulping Temperatures

R. W. COLLIER

From experiments on the degradation of hemicellulose in 7.0 and 10.0% NaOH solutions at 160 to 180°C., it was found that the amount of acid-precipitable material (beta-cellulose) decreased linearly with time of heating, a condition for zero-order kinetics. The reaction was shown to be in reality a pseudo zero-order reaction because the rate of degradation is dependent upon the initial concentration of beta-cellulose. The kinetic data are in agreement with the stepwise end-degradation mechanism whereby the reducing end-group is split off as a saccharinic acid leaving a new reducing group on the main fragment. The new reducing group reacts as before and the stepwise process continues. This appears to be the first instance where the kinetics of the alkaline degradation of a polysaccharide has been reported, and where kinetic data have been used to support the stepwise end-degradation mechanism. This work provides experimental evidence that the destruction of beta-celluloses in solution occurs by this mechanism under alkaline pulping conditions. Specific reaction rate constants and activation energies were calculated for the reaction at the two levels of sodium hydroxide studied.

In the course of studying the effect of hemicellulose on pulping, it became of interest to investigate the rate of degradation of slash pine hemicellulose in alkaline solutions at conventional pulping temperatures. Many workers have observed that cellulose and hemicelluloses are destroyed by heating in alkaline solutions. Richtzenhain and Abrahamsson (*1*) studied the destruction of beta-cellulose from spruce pulp and hemicellulose from spruce and beech woods by boiling in 0.5 *N* and 1 *N* NaOH. Similar studies on hemicellulose isolated from spruce and beech woods and from straw were made at higher temperatures by Prey and coworkers (*2*). An investigation of the destruction of the polysaccharides dissolved in 18% NaOH was carried out by Franzon and Samuelson (*3*) who determined the loss in beta-cellulose from solutions obtained by steeping a rayon grade spruce pulp and a cotton-derived hydrocellulose. These investigations all have in common the observations that the degradation is accompanied by a decrease in the D.P. and quantity of beta-cellulose, and by an increase in acid formation.

The mechanism of the alkaline degradation of carbohydrates was the subject of extensive investigations by Kenner and co-workers (*4*). Studies were made of the effect of lime water at room temperature on simple glycosides and the lower polysaccharides, including cellobiose and cellotetraose. In the degradation of reducing dissaccharides it was found that the glycosidic bond is broken, with the formation of one molecule of a saccharinic acid from the reducing end, and one molecule of sugar from the glycosidic portion. The elucidation of this mechanism and its extension to cellulose is discussed and illustrated by Richards (*5*) and Lindberg (*6*). The classical elimination of an alkoxyl group from the position beta to a carbonyl group is offered as explanation for cleavage of the glycosidic bond. The reaction is initiated by enolization of the reducing end-group in cellulose to the 2-oxo or fructose structure. This puts the carbonyl group in the position beta to the glycosidic bond in the required arrangement for elimination to take place. The glycosidic bond is ruptured with the main chain being eliminated as the cellulose anion. The diketo fragment from the reducing end-group is converted by a benzilic acid rearrangement to D-gluco-isosaccharinic acid. The cellulose chain with one less glucose residue is regenerated with a new reducing end-group, allowing the process to continue. This reaction, frequently referred to as a "peeling" process, is, therefore, seen to be a stepwise degradation from the reducing end.

The work of Corbett and Richards (*7*) shows that the theory applies to the degradation of cotton in 0.5 *N* NaOH at 170°C. To demonstrate that thermal degradation is not a factor, cotton was heated in a buffer solution (pH 7.2–7.4) at 170°C. Very little weight loss and only a slight drop in D.P. were observed. It was concluded that some alkaline chain scission occurs at the higher temperature to produce chains with new reducing end-groups which are capable of undergoing the stepwise degradation. It is this type of degradation which is responsible for the major weight loss (*8*).

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Evidence also has been presented that this degradative mechanism applies as well to sulfate pulping. Saarnio and Gustafsson (*9*) were able to recover only 2% of the polysaccharides removed from wood during a sulfate cook. The lost material is converted to acidic products. In a preliminary study of the acids in sulfate black liquor, Green (*10*) has isolated two saccharinic acids and obtained indications of others by paper chromatography.

EXPERIMENTAL

The purpose of the present work was to determine the kinetics of the alkaline degradation of hemicellulose at pulping temperatures. Alkaline solutions of hemicellulose, obtained by extracting a wood pulp, were heated for varying periods of time and the amount of acid-precipitable material (beta-cellulose) remaining was determined.

Preparation of Hemicellulose Solutions

Pulp Used. A rayon grade wood pulp was prepared in the pilot plant from slash pine chips which were given a mild prehydrolysis and pulped by the sulfate process. The pulp was bleached by chlorination and hypochlorite bleaching stages. It was washed and centrifuged to approximately 65% moisture content and was not dried further.

Extraction of Pulp. Pulp was steeped in a solution of rayon grade sodium hydroxide by making a slurry at 3% consistency. Nominally the caustic concentration was 10%, but a slight excess was used to allow for alkali lost due to sorption by the pulp. The steeping liquor was recovered by filtering the slurry on a Buchner funnel and using a rubber dam for final removal. Fifteen pulp samples were steeped in succession by reusing the liquor and adding strong make-up caustic between steeps to maintain the proper strength and volume. The hemicellulose stock solutions prepared in this manner contained 104 to 113 g. per l. of sodium hydroxide and 12 to 13 g. per l. of beta-cellulose. These solutions were kept refrigerated until used, which was not over 3 days. The stock solutions were diluted with caustic solutions or water to vary the concentrations of sodium hydroxide or hemicellulose as desired.

Kinetic Runs

Heating Procedure. Hemicellulose solutions (150 ml.) were placed in stainless steel autoclaves of one liter capacity and heated by immersion in a thermostatically controlled, electrically heated glycerine bath. The thermostat was capable of controlling the temperature to within ±1°C. Temperature was determined by a thermometer inserted into a well extending into the bath beneath the autoclaves. A calibration was made of the equipment by measuring the steam pressure generated by heating water in the autoclaves using a precision test gage. For the experimental runs no attempt was made to exclude air and the autoclaves were not vented during heating. Corbett and Richards (*7*) showed that the presence of ferric ions (from the autoclave) or atmospheric oxygen had little, if any, effect upon the rate of alkaline degradation of cellulose at 170°C. Four autoclaves could be heated simultaneously. After immersion in the preheated bath, it required approximately 15 min. to bring the autoclaves and contents to the desired temperature. Fifteen minutes was therefore allowed for heat-up time and the end of this period was considered to be the "zero" or starting time. One autoclave was removed at "zero" time and the others one by one at successively longer periods. Immediately after removal, each was immersed in a tub of cold water to quench the reaction.

Analysis of Hemicellulose Solutions. Samples of the hemicellulose solutions were analyzed for beta-cellulose content, before and after the kinetics runs, by a modification of Method CCA 10, Determination of Beta and Gamma Cellulose (*11*). In this method, organic material is determined by oxidation with potassium dichromate in strong acid solution, and the amount of oxidant consumed is quantitatively determined by back-titration with ferrous ammonium sulfate. Important changes followed in the present work will be noted. Oxidation was carried out by heating the acidic dichromate solution containing the sample at reflux for 10 min. After cooling to room temperature the excess dichromate was electrometrically titrated with a Beckman Automatic Titrator, Model K. Beta-cellulose was determined by difference after precipitation with a slight excess of 6 N H_2SO_4. The precipitate was separated by filtering through a pad of Johns-Manville Hyflow Super-Cel on a coarse fritted glass crucible. The ferrous ammonium sulfate was standardized daily against a freshly prepared solution of high purity cotton linters dissolved in 72% H_2SO_4 (*12*). Results are, therefore, expressed in terms of an equivalent quantity of cellulose.

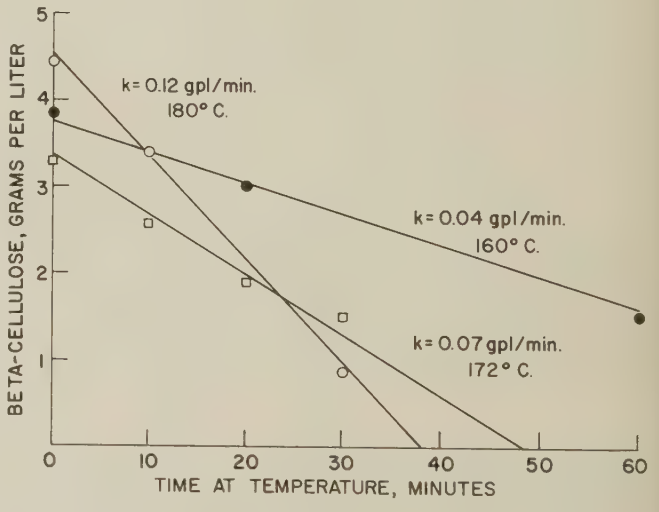

Fig. 1. Rates of degradation of beta-cellulose in 7% NaOH solutions

RESULTS AND DISCUSSION

The decrease of beta-cellulose in 7.0% NaOH solution at 160, 172, and 180°C. was found to be linear with time of heating (Fig. 1). This is a condition characteristic of a zero-order reaction for which the kinetics relationship is $-dc/dt = k$. The slope of the straight line is k, the zero-order specific rate constant. In all cases the slopes were calculated by the method of least squares.

There are only a limited number of mechanisms which could give zero-order kinetics in the system studied. A reaction occurring on a catalytic solid surface could show such an effect if the rate depended upon the area of the surface, such as the walls of the container. To investigate this effect, ceramic Berl saddles and 40-mesh stainless steel wire cloth were placed in separate autoclaves with hemicellulose solutions to increase the surface area in contact with the liquid. Starting with a beta content of 4.8 g. per l. in 7.0% NaOH, heating was carried out for 15 min. at 170°C. For all samples, including a control, the beta content after heating was between 2.24 and 2.47 g. per l. indicating that the degradation is not a surface-catalyzed reaction.

To clarify the factors governing the kinetics of beta-cellulose degradation, it is helpful to make a comparison with a simpler carbohydrate system. According to the degradation mechanism previously discussed, the disappearance, or consumption, of a disaccharide should follow first-order kinetics; its rate of disappearance should be proportional to its instantaneous concentration. In typical fashion the concentration by weight will decrease at the same rate as the *number of molecules* of reactant decreases and a decrease in rate will be observed as reactant is consumed. It will be different, however, for a polysaccharide reacting by the same mechanism. In this case a new reducing end-group is formed for each group lost, and there is no change in the number of chain molecules. According to the mechanism, the reaction theoretically is first-order with respect to the polysaccharide, but the usual decrease in rate is prevented from taking place and the degradation, properly, should be called a pseudo zero-order reaction. If the course of the reaction is followed by determining the change in beta-cellulose content, a decrease in weight will occur, but there will be essentially no change in the number of molecules reacting. Actually the number of chain molecules classified as beta-cellulose decreases very slowly, but for the purpose of discussion this may be disregarded. Therefore, under a given set of conditions, the degradation will proceed at a fixed rate which is dependent upon the number of molecules *originally* present. The rate is thus a function of the original, but not the instantaneous concentration of beta-cellulose. The rate also would be a function of the D.P. if a fixed concentration by weight were considered, and would be a function of the beta-cellulose content if a given D.P. were taken.

Consequently, experiments were carried out to determine the effect of the initial concentration of beta-cellulose upon the rate of degradation. Stock solutions of hemicellulose were diluted with pure caustic of the same strength (10.0%) to give beta-cellulose contents, on a relative basis, of roughly 1.0, 0.8, 0.6, and 0.4. These resulted in initial concentrations of 12.3, 10.8, 7.7, and 5.9 g. per l. This method of preparation provided samples containing material of the same D.P. distribution, differing only in concentrations of hemicellulose. The results of kinetic runs, presented in Fig. 2, show that the rate is dependent upon the initial concentration. Runs at the 1.0 and 0.6 levels were performed in duplicate and the slopes were found to be statistically different at the 95% confidence level.

With the concentration dependence established, the kinetic data can be calculated in another form. The

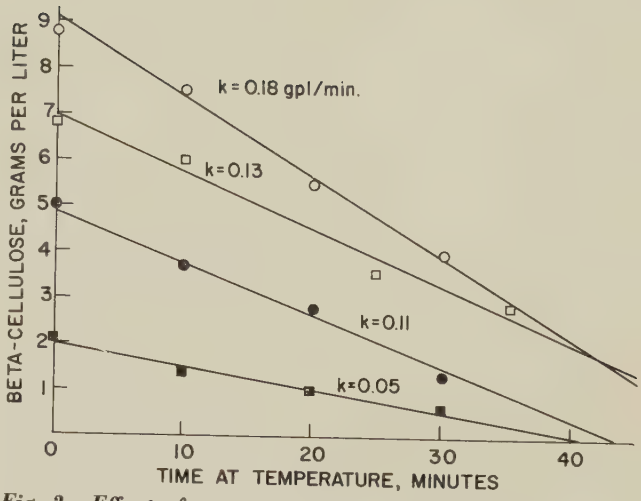

Fig. 2. Effect of concentration of beta-cellulose on rates of degradation in 10% NaOH at 170°C.

rate expression, $-dc/dt = k$, can be changed to correct for concentration dependence by substituting $k = k'C_0$, where C_0 is the concentration at "zero" time. The revised expression is $-dc/dt = k'C_0$, the kinetic form for a first-order reaction. In a sense then, k' is the first order specific rate constant and is independent of the initial and instantaneous concentrations. Values for k' were calculated from the data used in Figs. 1 and 2 and are summarized in Table I.

Table I. Rate Constants Corrected for Concentration Dependence

Temperature °C.	NaOH, %	k', min.$^{-1}$
160	7.0	0.010
172	7.0	0.021
180	7.0	0.027
170	10.0	0.021[a]
180	10.0	0.031

[a] Av. of four, data from Fig. 2.

Activation energies were calculated using the k' values in Table I. An Arrhenius plot is shown in Fig. 3 for the experiments with 7.0% NaOH solutions. The activation energy derived from this plot was calculated to be 19.8 kcal. per mole. Since only two sets of data were obtained for degradation in 10% NaOH, the integrated form of the Arrhenius equation was used, and the activation energy was calculated to be 15.5 kcal. per mole.

The effect of NaOH upon the rate of degradation was not studied explicitly. Because of the high concentrations of sodium hydroxide used in these experiments, changes in this component were negligible during the course of reaction, and its effect upon the order of reaction would not be evident. Likewise, if a salt effect were important, it would not be possible to tell under the conditions studied. It appears, however, from a comparison of the rate constants in Table I, that the rate of degradation is increased by raising the concentration of sodium hydroxide. At the same temperature, slightly higher rate constants are indicated for 10.0% than for 7.0% NaOH. One other aspect of the effect of alkali can be mentioned. Kenner and Richards (*13*) suggested that lime water, specifically $(CaOH)^+$, acted as a catalyst. No evidence for a catalytic effect

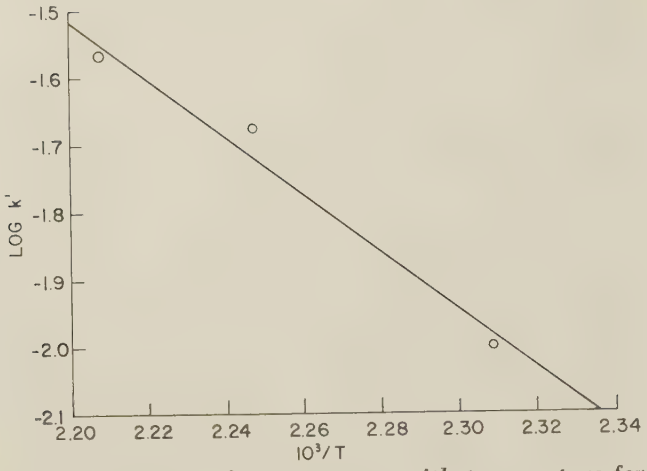

Fig. 3. Variation of rate constant with temperature for degradation of beta-cellulose in 7% NaOH

was observed, however, from an experiment conducted with 10% NaOH solution saturated with respect to calcium hydroxide.

CONCLUSIONS

It was found that the degradation of hemicellulose in alkaline solutions at high temperatures exhibits pseudo zero-order kinetics as determined by the decrease in beta-cellulose content. This is consistent with a mechanism proposed by others (*4*, *5*, *6*) in which the reaction proceeds by stepwise elimination of the end-groups with the production of new reducing end-groups so that the process continues. These findings give support for the proposed mechanism, and give experimental evidence that under alkaline pulping conditions hemicelluloses are destroyed by the stepwise end-group degradation. The rate of degradation appears to be increased by increasing the concentration of sodium hydroxide.

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Transparentizing of Paper

FRANS V. E. VAURIO

A limited review of the literature on the transparentizing of paper indicates a need for more fundamental study. Most of the information on the subject is contained in the patent literature. The nature of the patents which have been issued indicates that, for the most part, an Edisonian approach has been followed. Transparency is defined as being characteristic of a substance which permits parallel rays of light to pass through it without being scattered. The transparentizing effect on paper by various liquids encompassing a wide range of refractive indices was studied. The criterion taken for transparency was the transparency ratio or the ratio of the amount of parallel light transmitted to the total amount of light transmitted. The tests were made on treated sheets sandwiched between Mylar film using the Glassine Association Transparency Meter. A definite peak in the transparency of filter paper was noted when treated with a liquid with an index of refraction of about 1.55 to 1.56. Since cellulose is birefringent, the index of refraction of the optimum transparentizing agent will be a compromise between the refractive indices.

Transparentizing of paper is of interest in many paper applications such as photo reproduction papers, paper lamp shades, tracing papers, window envelopes, bread wrappers, and other packaging applications.

Paper in its most common forms does not have good transparency without further treatment largely because of its fibrous nature and the consequent tendency for light to be scattered within it. It has been accepted that materials we describe as transparent have the ability to transmit parallel rays of light without scattering them seriously. Thus, we are able to see objects more distinctly through a material depending on the degree of transparency.

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REVIEW OF THE LITERATURE

A brief review was made of the technical and patent literature related to the transparentizing of paper. Theoretically, paper may be transparentized by treating it with a material which has an index of refraction equal to that of the fiber of which it is composed. A list of commercial products which might be useful in controlling the transparency of paper through the use of treatments other than those used in the manufacture of glassine or vegetable parchment was prepared, giving the index of refraction values in ascending order. These values are given in Table I. It should be noted that much of the information is taken from the technical

Table I. Refractive Indices for Various Chemicals (In Order of Increasing Index of Refraction)

Refractive index	*Temperature, °C.*	*Substance*	*Source*	*Reference no.*
1.4028	20	Methyl "Cellosolve"	Union Carbide Chemicals Co.	(*1*)
1.4058	25	Dowanol 8	Dow Chemical Co.	...
1.4062	20	Bunco (*n*-butyl isocyanate)	The Carwin Co.	...
1.408	20	Amyltriethoxy silane	Linde Air Products Co.	...
1.4148	20	Diethyl ethyl phosphonate	Virginia Carolina Chemical Corp.	...
1.42–1.43	20	Dow Corning 510 Fluid	Dow Corning Co.	...
1.421	20	Butyl methacrylate monomer	Rohm and Haas Co.	...
1.424	20	Tributyl phosphate	Monsanto Chemical Co.	...
1.4269	25	Dimethyl formamide	Du Pont Co.	...
1.428–1.429	83	Armor wax 112	Pennola Oil Co.	...
1.434	25	KP140 (tributoxyethyl phosphate)	Ohio-Apex Division, Food Machinery & Chem. Corp.	...
1.4358	20	Tri-*n*-butyrin	Tennessee Eastman	...
1.438	25	Citroflex A2 (acetyl triethyl citrate)	Charles Pfizer Co.	(*1*)
1.440	25	Citroflex 2 (triethyl citrate)	Charles Pfizer Co.	...
1.441	25	Citroflex A4 (acetyl tributyl citrate)	Charles Pfizer Co.	...
1.441	25	Citroflex A8 (acetyl tri-2-ethylhexyl citrate)	Charles Pfizer Co.	...
1.442	25	Butyl stearate	Ohio-Apex Division, Food Machinery & Chem. Corp.	...
1.443	25	Citroflex 4 (tributyl citrate)	Charles Pfizer Co.	...
1.4446	25	Dibutyl fumarate	Monsanto Chemical Co.	...
1.446	25	Adipol 2EH [di(2-ethylhexyl) adipate]	Ohio-Apex Division, Food Machinery & Chem. Corp.	...
1.4475	20	Diethylene glycol	Allied Chem. & Dye Corp.	...
1.4481	20	Bis (2-ethylhexyl) 2-ethylhexyl phosphonate	Virginia Carolina Chemical Corp.	...
1.450	25	Adipol XX (diisodecyl adipate)	Ohio-Apex Division, Food Machinery & Chem. Corp.	...
1.45	20–22	Kerosene	.....	(*2*)
1.45–1.48	25	Uniphat 434M	General Mills	...
1.4502	20	Cyclohexanone	.....	(*3*)
1.4505	20	Tetrahydrofurfuryl alcohol	Quaker Oats	...
1.4525	25	Drapex 3.2	Argus Chemical Corp.	...
1.4528	20	Advawet X212	Advance Solvents and Chem. Corp.	...
1.462	20	Cyclohexanol	Monsanto Chemical Co.	...
1.4621	20	2-Methoxymethyl 2,4-dimethyl pentanediol-1,5	Union Carbide Chemicals Co.	...
1.4655	20	Indopol polybutene L-10	Indoil Chemical Co.	...
1.4656	20	Cyclohexandiol	.....	(*3*)
1.466	20	Diallyl itaconate	Charles Pfizer Co.	...
1.4719	25	Paraplex G-60	Rohm and Haas Co.	...
1.4729	25	Glycerol	.....	(*3*)
1.4733	20	Dimethylsulfolane	Shell Development Co.	...
1.475	20	Bis (β-chloroethyl) vinyl phosphonate	Monsanto Chemical Co.	...
1.4759	20	Indopol polybutene L-100	Indoil Chemical Co.	...
1.4779	20	Linseed oil (Superior)	Kellogg	...
1.480	20	Castor oil	.....	(*1*)
1.4830	25	Santicizer 601	Monsanto Chemical Co.	...
1.4835	25	Santicizer 602	Monsanto Chemical Co.	...
1.4840	25	Dioctyl phthalate	Monsanto Chemical Co.	...
1.4844	25	Di-isodecyl phthalate	Monsanto Chemical Co.	...
1.486	20	Furfuryl alcohol	The Quaker Oats Co.	...
1.486	20	Di-2-ethylhexyl phthalate	Ohio-Apex Division, Food Machinery & Chem. Corp.	...
1.4870	20	Trischloroethyl phosphite	Westvaco Mineral Products	...
1.488	20	Bis (β-chloroethyl) β-chloroethyl phosphonate	Monsanto Chemical Co.	...
1.488	20	Tris (β-chloroethyl) phosphite	Monsanto Chemical Co.	...
1.489–1.490	20	Energetic W-100	Armour and Co.	...
1.490	20	Santicizer B-16	Monsanto Chemical Co.	(*3*)
1.490	25	Di-*n*-butyl phthalate	Ohio-Apex Division, Food Machinery & Chem. Corp.	...
1.4915	25	Dibutyl phthalate	Monsanto Chemical Co.	...
1.4918	20	Indopol polybutene H-100	Indoil Chemical Co.	...
1.492	25	Di-"Carbitol"-phthalate	Ohio-Apex Division, Food Machinery & Chem. Corp.	...
1.4942	20	Sym-tetrachlorethane	.....	(*3*)
1.4955	20	Indopol polybutene H-300	Indoil Chemical Co.	...
1.4963	20	Chloromethyl phosphonic dichloride	Monsanto Chemical Co.	...
1.497	20	MPS 500 (methyl pentachlor stearate)	Hooker Electrochemical Co.	(*3*)
1.498	25	Santicizer E-15 (monoethyl phthalate ethyl glycollate ester)	Monsanto Chemical Co.	...
1.4984	25	Benzoflex P-600	Tennessee Products and Chemicals Co.	...
1.4993	25	Paraplex P444	Rohm and Haas Co.	...
1.500	25	Methox (dimethoxyethyl phthalate)	Ohio-Apex Division, Food Machinery & Chem. Corp.	...
1.500	25	Santicizer 3 (*n*-ethyl-*p*-toluenesulphonamide)	Monsanto Chemical Co.	...
1.5002	25	Diethyl phthalate	Monsanto Chemical Co.	...
1.5011	20	Benzene	.....	(*3*)
1.5016	20	Polyrad 0515	Hercules Powder Co.	...
1.502	20	Retal	Newport Industries	...
1.502	20	Stabilizer 17M	Advance Solvents & Chemical Corp.	...
1.5025	25	β,β′-Thiodipropionitrile	Monsanto Chemical Co.	...
1.504	25	Santicizer M17 (monomethyl phthalate ethyl glycollate ester)	Monsanto Chemical Co.	...
1.505	25	Chlorwax 40	Diamond Alkali	(*3*)
1.5054	20	*o*-Xylene	.....	(*3*)

Table I. Refractive Indices for Various Chemicals (*Continued*)

Refractive index	*Temperature, °C.*	*Substance*	*Source*	*Reference no.*
1.5055	20	Tetrachloroethylene	Du Pont	...
1.506	20	Santicizer 180	Monsanto Chemical Co.	(3)
1.507–1.510	25	Santicizer 141 (2-ethyl-hexyl diphenyl phosphate)	Monsanto Chemical Co.	(3)
1.515	20–22	Cedar wood oil	.....	(2)
1.516	20	Polyrad 0100	Hercules Powder Co.	...
1.5169	20	Polyrad 0200	Hercules Powder Co.	...
1.5173	20.6	Anisole	.....	(2)
1.5184	20	Hercolyn (methyl ester of abietic acid)	Hercules Powder Co.	...
1.5194	20	Diallyl phthalate (monomer)	Shell Development Co.	...
1.520	30	Harflex 90. (Dibenzylsebacate)	W. C. Hardesty Co.	(3)
1.521	20	Petrex T7HT	Hercules Powder Co.	...
1.5211	20	Methyl phenylcarbinol	.....	(3)
1.523	25	Phenyl cyclohexane	Monsanto Chemical Co.	...
1.5236	25	*o*-Chlorotoluene	.....	(1)
1.524	20	Phenyltrichlorsilane	Linde Air Products Co.	...
1.5246	20	Chlorobenzene-mono	.....	(3)
1.5249	15	Trimethylene bromide (1,3-dibromopropane)	.....	(1)
1.5251	20	Dowanol 17	Dow Chemical Co.	...
1.5252	25	Benzoflex P-200 (polyethylene glycol 200 dibenzoate)	Tennessee Products and Chemicals Co.	...
1.526	20	Furfural	Quaker Oats Co.	...
1.528	20	Abitol (hydroabietyl alcohol)	Hercules Powder Co.	...
1.5283	20	Benzoflex P200	Tennessee Products and Chemicals Co.	...
1.529	20	Abalyn (methyl ester of rosin)	Hercules Powder Co.	...
1.5340	20	Acetophenone	.....	(3)
1.535	25	Nevillac OA	Neville Chemical Co.	...
1.537	25	Benzoflex P300	Tennessee Products and Chemicals Co.	...
1.5379	20	Ethylene bromide	.....	(2)
1.538	20	Santicizer 160 (butyl benzyl phthalate)	Monsanto Chemical Co.	(3)
1.538	25	Dimethyl phthalate	Monsanto Chemical Co.	...
1.5382	20	Panapol 2C	Pan American	...
1.5386	20	Dowanol 1 (phenyl "Cellosolve")	Dow Chemical Co.	...
1.5399	20	Benzyl alcohol	.....	(3)
1.540	20	Santicizer 8 (*o*- and *p*-toluenesulfonamide)	Monsanto Chemical Co.	(3)
1.5410	20	Rosin amine D	Hercules Powder Co.	...
1.5439	20	Tetralin	.....	(3)
1.5440	20	Benzaldehyde	.....	(1)
1.545	25	Nevillac OP	Neville Chemical Co.	...
1.547	20	KP504 [Bis (dimethylbenzyl) carbonate]	Ohio-Apex Division, Food Machinery & Chem. Corp.	(3)
1.5470	..	HB20	Monsanto Chemical Co.	...
1.5474	20	*o*-Nitrotoluene	.....	(2)
1.5495	20	Indonex 632-$^1/_2$	Indoil Chemical Co.	...
1.550	60	Triphenyl phosphate	Monsanto Chemical Co.	...
1.5518	22	*o*-Dichlorobenzene	.....	(1)
1.5523	20	Nitrobenzene	.....	(3)
1.553	25	Bis (dimethylbenzyl) ether	Ohio-Apex Division, Food Machinery & Chem. Corp.	...
1.5535	20	Anethole, technical	Hercules Powder Co.	...
1.555	25	Kronitex AA (tricresyl phosphate)	Ohio-Apex Division, Food Machinery & Chem. Corp.	...
1.556	20	Panaflex BN-1	Pan American Petroleum and Transport Co.	(3)
1.5571	20	Tri-*o*-cresyl phosphate	Eastman Chemical Co.	...
1.5586	20	Tricresyl phosphate	Monsanto Chemical Co.	...
1.560	25	Santicizer 140 (cresyl diphenyl phosphate)	Monsanto Chemical Co.	...
1.560	25	Nevillite No. 123	Neville Chemical Co.	...
1.5604	20	Bromobenzene	.....	(2)
1.564	20	Indonex (dark hydrocarbon)	Indoil Chemical Co.	...
1.567	27	R108 Phenolic coating intermediate	General Electric Co.	...
1.5675	25	HB40 (partially hydrogenated mixture of isomeric terphenyl)	Monsanto Chemical Co.	...
1.5688	20	*o*-Toluidene	.....	(1)
1.572	74	Diphenyl phthalate	Monsanto Chemical Co.	...
1.5808	25	Isopropylbiphenyl	.....	(1)
1.582	20	Dow 276-V2	Dow Chemical Co.	(3)
1.582–1.590	..	*o*-Xenyl diphenyl phosphate	.....	(3)
1.5913	20	Methyl-*o*-benzoyl benzoate	.....	(3)
1.596	25	Nevinol	Neville Chemical Co.	(3)
1.597	25	Nevillac 10 (PHO)	Neville Chemical Co.	...
1.5980	19	Bromoform	.....	(2)
1.599	25	Nevillac TS	Neville Chemical Co.	...
1.604	20	Resin 622	Dow Chemical Co.	...
1.609	20	*o*-Iodotoluene	.....	(2)
1.613	25	*o*-Nitrobiphenyl (supercooled)	Monsanto Chemical Co.	...
1.621	18	Iodobenzene	.....	(1)
1.621–1.627	20	Aroclor 1242 (chlorinated biphenyl)	Monsanto Chemical Co.	(3)
1.6283	20	Quinoline	.....	(1)
1.6315	15	Carbon disulphide	.....	(1)
1.63321	20	α-Monochloronaphthalene	.....	(1)
1.638	20	*sym*-tetrabromoethane	.....	(1)
1.66011	16.5	α-Bromonaphthalene	.....	(1)
1.7425	15	Methylene iodide	.....	(1)

Note: The refractive index is given for the sodium D line at 20°C. unless otherwise noted.

literature furnished by the suppliers and represents the latest information available. These index of refraction values may differ appreciably from those for chemically pure products or for technical grades from other sources. However, it was felt that the commercially available materials would serve the purpose intended and might also serve as a reference list for development purposes. The names of the suppliers are given whenever possible. Some of the index of refraction values were obtained from sources other than the supplier and are so noted (*1–4*).

Table II lists the index of refraction for a number of resins and polymers which might be considered for controlling the transparency of paper. Some of these might be applied in the monomeric form and others from emulsions or solutions.

Paper may be transparentized during the course of its manufacture as in the case of glassine and vegetable parchment. This involves beating or chemically treating the fibers until they become plastic and can be used to form a dense sheet which becomes transparent when subjected to the consolidating action of the supercalender. Such a sheet, unless plasticized, is brittle and unable to withstand folding or creasing. The process of making glassine is relatively slow because of the time required in the beating process and also because of the slowness of the rate at which water drains from the highly hydrated fibers. The manufacture of parchment is also relatively slow due to the need for careful rinsing required to remove the acid used in treating the fiber.

Thus, it becomes of interest to achieve transparency in paper by treating it with some material which does not detract from its end use requirements. Theoretically, this would mean filling the voids and interstices of the paper with a transparent substance with an index of refraction the same as that of the paper.

Table II. Refractive Indices of Various Solids

Refractive index	*Temperature, °C.*	*Product*	*Source*	*Reference*
1.3	20	Teflon	Du Pont Co.	(*4*)
1.450	20	Atlas G750	Atlas Powder Company	...
1.460	20	Alvar	Shawinigan Resins Corp.	...
1.480	20	Atlas G749	Atlas Powder Company	...
1.480	20	Atlas G797	Atlas Powder Company	...
1.480	20	Half-second cellulose acetate butyrate	Eastman Chemical Products Co.	...
1.489	20	Acryloid B-72	Rohm and Haas Co.	...
1.491–1.50	20	Methyl methacrylate polymer	Du Pont Co.	...
1.492	20	Epoxide 201	Union Carbide Chemicals Co.	...
1.5002–1.5022	20	Staybelite resin	Hercules Powder Co.	...
1.505	23	Chlorinated paraffin	.....	(*3*)
1.51	20	Polyvinyl alcohol	.....	(*4*)
1.510–1.515	20	Atlas G678	Atlas Powder Co.	...
1.514	20	Petrex 7HT	Hercules Powder Co.	...
1.52	20	Polybutadiene	.....	(*4*)
1.52	20	Polyisopropenyl methyl ketone	.....	(*4*)
1.5213	20	Perbunan	.....	(*4*)
1.5225–1.5230	20	Staybelite ester No. 2	Hercules Powder Co.	...
1.525	20	Methyl rubber	.....	(*4*)
1.5266	69.9	*p*-Dichlorobenzene	.....	(*2*)
1.5269	20	Neolyn 40	Hercules Powder Co.	...
1.529	20	Petrex 130H	Hercules Powder Co.	...
1.530	20	Polyvinyl chloride-acetate	.....	(*4*)
1.530	20	Nylon	.....	(*4*)
1.531	20	Neolyn 20	Hercules Powder Co.	...
1.532	20	Estergum 8L	Hercules Powder Co.	...
1.532	20	Pentalyn H	Hercules Powder Co.	...
1.536	20	Flexalyn	Hercules Powder Co.	...
1.540	20	Polyvinyl chloroacetate	.....	(*4*)
1.543	20	Flexalyn C	Hercules Powder Co.	...
1.544	20	Polypale resin	Hercules Powder Co.	...
1.544	20	Polyvinyl chloride	.....	(*4*)
1.544	20	Pentalyn A	Hercules Powder Co.	...
1.545	20	Abietic acid	Hercules Powder Co.	...
1.545	20	Staybelite ester No. 1	Hercules Powder Co.	...
1.545	20	Polypale esters No. 1 and 2	Hercules Powder Co.	...
1.5453	20	Rosin	Hercules Powder Co.	...
1.546	20	Staybelite ester No. 10	Hercules Powder Co.	...
1.546	20	Polypale ester No. 10	Hercules Powder Co.	...
1.55	20	Maraglas No. 655	Marblette Corp.	...
1.5512	20	*n*-Polychloroprene	.....	(*4*)
1.558	20	Neoprene	.....	(*4*)
1.564	20	Copolymer of styrene and maleic anhydride	.....	(*4*)
1.57	20	Dow latex 512K	Dow Chemical Co.	...
1.571	25	Dow polystyrene resin PS-1,-2,-3	Dow Chemical Co.	...
1.64	25	Mylar	Du Pont Company	...

The index of refraction of papermaking fibers apparently has not been studied as extensively as those of textile fibers.

We should expect three different indices for cellulose fibers which can be considered optically biaxial crystals according to Hermans (*5*). However, only two have been given any practical consideration. Hermans studied model fibers of regenerated cellulose using the the polarizing microscope. The index of refraction for one plane of polarization was found to vary from 1.506 to 1.534 and for the other plane from 1.541 to 1.574,

depending on the moisture content (which ranged from from 0.198 to 0.023 g. of water per gram of cellulose).

Preston (*6*) found that cellulose fibers from cotton are birefringent, having an index of refraction of 1.578 in the axial direction of the fiber and 1.532 in the transverse direction. He used the Becke method in which a bright line outlining the fiber immersed in a liquid is observed. If the liquid has a lower index of refraction than the fiber, the Becke line moves toward the fiber when focusing up from sharp focus. Haywood (*7*) gives 1.50 as the index of refraction for cellulose. The original source for this information is not given.

The patent literature relating to the transparentizing of paper is relatively meager and much of it stems from nontechnical development. However, information on the various agents claimed to be useful, in the patent and other literature, may be of interest.

A mixture containing petroleum and a resinlike gum dammar, copal, or colophony with a trace of vegetable oil like castor oil, poppyseed oil, walnut oil, or resin oil for transparentizing paper was patented by Allott (*8*).

According to Holzberger (*9*), Damar, Manila, and Sandarac resins will transparentize paper.

A mixture of methyl abietate and sucrose octaacetate in ethylene dichloride was used as a transparentizing agent by Davidson (*10*).

William F. Gresham (*11*), in his patent covering new polymers, mentions that the linear polyformal resins with indices of refraction in the range of 1.500 to 1.548 (25°C.) should be of value in transparentizing paper.

Robert R. Parmentier (*12*) suggests using mineral oil in a solvent mixture and then sealing the mineral oil in the paper with a solution containing wax.

Spurlin (*13*) claims that a plastic with an index of refraction of 1.50 to 1.52 is needed to transparentize paper and cites formulas containing nitrocellulose and tricresyl phosphate for this purpose.

Swett (*14*) treated one side of glassine envelope window paper with a calcium chloride solution to produce a mat finish to cut down glare, and the other side with tannic acid dissolved in alcohol with or without terpin hydrate to improve transparency.

Schwalbe, Fuchs, and Schnecko (*15*) suggest making tracing paper by coating paper with a nitrocellulose lacquer plus fillers like chalk, starch, and talc.

Finzel and Drew (*16*) used several methods to produce a transparent paper. These included treating paper with a solution of paraffin and ester gum in a solvent mixture of naphtha and alcohol, with a paraffin solution in toluene followed by a nitrocellulose lacquer, or with a soap solution followed by a wax impregnation.

Gabriel (*17*) coated paper with a cellulose nitrate or cellulose acetate lacquer containing a nonvolatile plasticizer like glycerol butyl phthalate or glycerol benzyl phthalate.

Aycock and Kelley (*18*) patented their method of transparentizing paper with a high molecular weight alcohol or fatty acid monoglyceride and a methylated or partially methylated methylol urea.

The Indoil Co. suggests in their advertising that their polybutenes can be considered for making tracing paper.

Hinman (*19*) claims the use of white mineral oil, wood rosin, abietic acid, hydrogenated rosin, ester gum, castor oil, chlorinated diphenyl, coconut oil, and paracoumarone indene resin for making tracing paper.

Vincent (*20*) lists vegetable oil, waxes, ethyl abietate and cellulose hydrate as treatments for tracing paper.

A description of the manufacture of window envelopes and a discussion of the chemistry of varnishes made up of oils and resins used in forming window envelopes is given by Papineau-Couture (*21*).

Hermanson (*22*) made a transparent sheet from synthetic fibers and a transparent polymerized resin.

Hugo Klein (*23*) lists mineral oil, balsam, castor oil, peanut oil, natural gums, resins, rosin derivatives, sorbitol, dibutyl phthalate, tricresyl phosphate, and sucrose octaacetate as treating agents for paper to render it transparent or translucent.

Knaggs and his co-workers (*24*) in making transparent paper used rag paper and treated it with an emulsion of oil and fat or wax containing gelatin. The paper was softened with glycerin or magnesium chloride and made water resistant by treating it with formaldehyde.

EFFECT OF TRANSPARENTIZING AGENT

The effect of the index of refraction of various liquids on the transparency developed in paper treated with the liquids was studied at The Institute of Paper Chemistry.

Procedure

The "transparency ratio" or the ratio of the parallel transmitted light to the total transmitted light was used as the criterion for the degree of transparency. According to Wicker (*25*) this relationship minimizes the influence of color on the measurement of transparency.

The Glassine Association Transparency Meter (*26*) was used in measuring the parallel and total transmittance of the treated paper. This instrument consists of a light source, lens, collimating tube, rheostats for adjusting the current produced by the Weston Photronic photocell which senses the amount of light transmitted, and a microammeter which indicates the photoelectric current.

An Abbé refractometer and a sodium vapor light were used in measuring the index of refraction of the various liquids.

Filter paper, Schleicher and Schull Co., New York, No. 589 Black Ribbon, 7 cm. diam. with an ash content below 0.01 mg. per circle was used in the experiments. The untreated paper had a total transmittance of 20.5% and the parallel transmittance was too low to measure with the Glassine Association Transparency Meter.

The liquids used in treating the papers were selected to represent a range of refractive indices, and included KP140 (tributoxyethyl phosphate), dioctyl phosphate, tributyl citrate, butyl stearate, tricresyl phosphate, Santicizer 140 (cresyl diphenyl phosphate), Dow Plasticizer Number 5 (*o*-xenyl diphenyl phosphate), 1-chloronaphthalene, α-bromonaphthalene, and glycerin. In order to increase the number of refractive index values, mixtures of liquid were also prepared.

In order to achieve complete penetration, the paper was conditioned over calcium chloride and then single sheets were subjected to a vacuum of about 2 mm. mercury with an oil pump. After about 5 min. under vacuum, the paper which was contained in a culture dish was then covered with liquid and the vacuum ended by admitting atmospheric air.

A buret with a plastic (Teflon, polytetrafluoroethylene) valve instead of the usual grease-lubricated glass stopcock was used to meter the transparentizing liquid to minimize the possibility of contamination of the organic liquid with stopcock grease.

A mercury closed-tube manometer was used to check the pressure during the vacuum treatment. Many of the liquids boiled profusely at the low pressures. Some fractional distillation probably occurred under these conditions causing some change in the refractive index of a given mixture.

After about 15 min., the sheet of paper was removed from the liquid and sandwiched in the fold of a piece of Mylar film. The Mylar film was 0.25×10^{-3} in. thick. The total and parallel transmission was measured using the Glassine Association Transparency Meter. Special care was taken to smooth out wrinkles and to work out any bubbles of air by massaging the sandwich between the thumb and fingers. The Mylar film served to furnish a fairly constant surface and to facilitate handling the oily paper.

A Sorenson Model 150 regulator and a 6-v. transformer were used to power the transparency meter. It was found that fluctuations occurred in the readings if the regulator were omitted. A magnetic-type regulator was found unsatisfactory. It is believed that a magnetic-type regulator accentuates fluctuations due to frequency variations.

The transparency meter was adjusted to give a zero reading when no light entered the photocell and to give a reading equivalent to 100% transmittance with the light on and with no sample. The sandwich of Mylar and treated paper was inserted first in the space between the collimating tube and the photocell housing and then in the space between the lens housing and the collimating tube and the meter values noted. The specimen was then removed from the Mylar sandwich and reinserted in the liquid in the culture dish.

The Mylar has an index of refraction of 1.64 according to Lanning (*27*) and has good transparency. A single thickness of dry Mylar had a total transmittance of 89.9% and a parallel transmittance of 84.0%. Videne, a Goodyear Tire and Rubber Co. polyester film, seems to have good clarity but was not available at the time this work was done.

Table III. Transparency Results—Transparency of Filter Paper Treated with Different Liquids

Index of refraction of liquid	*Liquid*	*Treatment time*	*Transmittance, % total*	*parallel*	*Transparency ratio, %*
1.4028	2-Methoxyethanol	15 min.	52.7	0.9	1.7
1.3962	2-Methoxyethanol	2 wk.	54.8	1.1	2.0
1.434	KP140 (Tributoxyethyl phosphate)	15 min.	56.5	1.0	1.8
1.4331	KP140 (Tributoxyethyl phosphate)	2 wk.	58.9	1.7	2.9
1.443	Citroflex 4 (Tributyl citrate)	15 min.	56.5	0.8	1.4
1.4428	Citroflex 4 (Tributyl citrate)	2 wk.	58.7	1.5	2.6
1.444	Butyl stearate	15 min.	59.5	1.9	3.2
1.4412	Butyl stearate	2 wk.	62.1	2.0	3.2
1.4729	Glycerol C.P.	15 min.	65.4	1.9	2.9
1.4575	Glycerol C.P.	2 wk.	69.0	2.5	3.6
1.464	1:1 Butyl stearate-dioctyl phthalate	15 min.	63.0	1.9	3.0
1.4609	1:1 Butyl stearate-dioctyl phthalate	2 wk.	66.8	2.3	3.4
1.4840	Dioctyl phthalate	15 min.	68.4	3.0	4.4
1.4838	Dioctyl phthalate	2 wk.	69.0	3.0	4.4
1.5026	3:1 Dioctyl phthalate-tricresyl phosphate	15 min.	71.7	3.1	4.3
1.4993	3:1 Dioctyl phthalate-tricresyl phosphate	2 wk.	70.0	3.7	5.3
1.5088	2:1 Dioctyl phthalate-tricresyl phosphate	15 min.	75.0	8.0	10.7
1.5229	2:1 Dioctyl phthalate-tricresyl phosphate	2 wk.	76.1	8.2	10.8
1.5213	1:1 Dioctyl phthalate-tricresyl phosphate	15 min.	73.0	6.1	8.4
1.5158	1:1 Dioctyl phthalate-tricresyl phosphate	2 wk.	75.5	6.3	8.4
1.5337	1:2 Dioctyl phthalate-tricresyl phosphate	15 min.	75.8	8.0	10.6
1.5275	1:2 Dioctyl phthalate-tricresyl phosphate	2 wk.	78.4	9.0	11.5
1.5399	1:3 Dioctyl phthalate-tricresyl phosphate	15 min.	69.1	9.0	13.0
1.5337	1:3 Dioctyl phthalate-tricresyl phosphate	2 wk.	79.0	10.9	13.8
1.5586	Tricresyl phosphate	15 min.	79.0	13.1	16.6
1.5533	Tricresyl phosphate	2 wk.	82.5	13.5	16.4
1.560	Santicizer 140 (Cresyl diphenyl phosphate)	15 min.	77.4	11.5	14.9
1.5593	Santicizer 140 (Cresyl diphenyl phosphate)	2 wk.	80.0	11.7	14.7
1.582	Dow Plasticizer No. 5 (*o*-Xenyl diphenyl phosphate)	15 min.	74.0	5.2	7.0
1.5979	Dow Plasticizer No. 5 (*o*-Xenyl diphenyl phosphate)	2 wk.	75.0	5.2	6.9
1.621	1:1 Dow Plasticizer No. 5-1-chloronaphthalene	15 min.	70.0	3.9	5.6
1.6144	1:1 Dow Plasticizer No. 5-1-chloronaphthalene	2 wk.	70.8	3.5	4.9
1.6317	1-Chloronaphthalene	15 min.	67.1	2.1	3.1
1.6305	1-Chloronaphthalene	2 wk.	66.1	2.7	4.1
1.66011	α-bromonaphthalene	15 min.	62.4	1.9	3.0
1.6555	α-bromonaphthalene	2 wk.	60.2	2.0	3.3

Note: The index of refraction values given for the initial transparency tests are those taken from the technical literature or calculated for the mixtures from these values. The index of refraction values for the tests after the 2-wk. treatment are those observed with sodium light at 73°F. at 50% R.H. on samples of the liquid in contact with the paper.

The specimens were held for about two weeks at 73°F. and 50% R.H. in the culture dishes which contained the liquid used in treating the paper and then retested for transparency. Some changes in the index of refraction of the liquids probably took place as a result of possible moisture pickup by the exposure to this humidity in the covered but unsealed culture dishes. Also there may have been some evaporation of the more volatile component from some of the mixtures to cause a shift in the index of refraction. The index of refraction was therefore tested after the two-week conditioning time by taking a sample from each culture dish.

The transparency ratio was calculated from the relationship for each of the specimens.

$$\text{transparency ratio} = \frac{\text{parallel transmittance}}{\text{total transmittance}} \times 100$$

RESULTS AND DISCUSSION

The results are given in Table III. The initial index of refraction shown is that taken from the manufacturers' or suppliers' literature or from standard references for the single component liquids and calculated for the mixture. The index of refraction values shown for the specimens which were treated for two weeks are observed values on samples of the liquids in the culture dishes. Any differences observed may be due to the reasons previously cited.

The greater variability shown in the results after 15 minutes of treatment may be due to a number of factors. The use of the index of refraction values from the technical literature is one. The differences in viscosity of the liquids may be an important factor for tests made so soon after treatment. The necessity to smooth out wrinkles and to work out bubbles made timing somewhat variable. The initial tests were made in an unconditioned laboratory and temperature variations may have introduced minor variations in index of refraction. The negative temperature coefficient for organic liquids of the type used was of the order of 0.0004 to 0.0005 per degree centigrade, i.e., a rise of 1°C. in temperature will result in a 0.0004 to 0.0005 lowering in index of refraction. This coefficient is different for the different types of liquids. We have no information on the temperature coefficient for paper. The surface effect was not completely eliminated by the use of the Mylar.

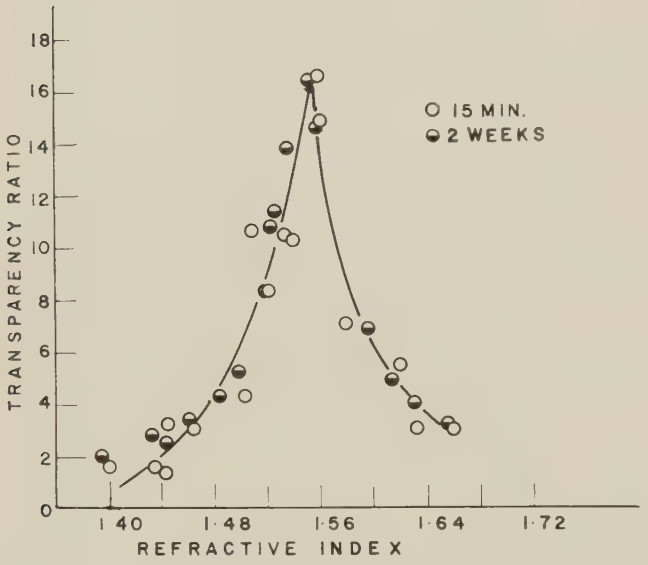

Fig. 1. Transparentizing paper. Transparency as a function of the index of the impregnating fluid

The results are plotted in Fig. 1, showing the transparency ratio as a function of the index of refraction.

The results may be used to establish an average index of refraction for this particular paper. We may observe that the peak in the curve occurs at an index of refraction range of approximately 1.553 to 1.560. This index of refraction range appears to be reasonable and consistent with known facts concerning cellulose as it exists in cotton and wood. Preston (*28*) attributes the differences in the two refractive indices in cellulose fibers to the spiral micellar structure. According to the Encyclopedia of Chemical Technology (*29*) the axial index of refraction of cotton cellulose is 1.578 and the transverse index is 1.532. According to Sherman and Sherman (*30*) dry cotton at 20°C. has 1.580 and 1.533 for the respective two refractive indices. The average index of refraction found for the filter paper used in this work agrees favorably with the average of the axial and transverse indices given above.

We may then generalize and say that materials which are potential transparentizing agents for this paper should have an index of refraction in the neighborhood of 1.553 to 1.560. The transparentizing ability drops off precipitously as we go higher or lower in index of refraction. Other types of paper may be expected to differ somewhat due to differences in chemical makeup.

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Variation in Cellulose Content of Douglas-Fir*

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Standard samples from eight Douglas-fir trees were analyzed for Cross and Bevan cellulose; in addition, a single tree of the group was analyzed from pith to bark. Lower cellulose yields were found in the core wood than in the outer wood of the single tree. Most of the variation in cellulose caused by age was found in the first 15 annual rings from the pith. Among the eight trees sampled, Cross and Bevan cellulose yields varied from 54.9 to 65.4 %. Corresponding water-resistant carbohydrate and alpha cellulose values ranged from 71.4 to 77.0% and 45.6 to 51.6%, respectively. Crown class, site, radial position, growth rate, and per cent summerwood failed to account for the variation observed in cellulose content, so that the difference between trees was ascribed to variability that is probably genetically controlled. The desirability of propagating trees with high cellulose content is discussed briefly.

It is of interest to both the forester and the pulp and paper mill operator to know if the cellulose content of trees can be regulated. Zobel and McElwee (*1*) have demonstrated that rather wide variations can exist between individual loblolly pine trees under the same conditions of growth and also showed that within single trees, there is a pattern of increasing cellulose content from wood near the pith to wood near the bark. This study was undertaken to establish the nature of the variation in cellulose content to be expected in Douglas-fir. The data obtained were analyzed to determine the effect of several growth factors on cellulose content.

METHOD

Eight Douglas-fir trees from two permanent sample plots at the University Research Forest near Haney, B. C. were selected for study. One plot was located on a good site (sword-fern type, site index 180 ft. at 100 yr.), and the other plot represented a poor site (lichen type, site index 80 ft. at 100 yr.). Tree ages ranged from 70 to 85 yr. The two largest and two smallest trees on each plot, representing dominant and intermediate crown classes, were felled, and cross-sectional disks removed at the breast-height level. Data for the trees are given in Table I.

Two radial positions within each tree were selected for preparation of analytical samples. One area (rings 16 to 25 from pith) lay completely in the heartwood, while the other included the five outer growth rings of sapwood. The largest tree (no. 2) was sampled continuously from pith to bark by dissecting the first five annual rings, then removing decadal samples up to an age of 75 yr., leaving the remaining six outer growth rings in the final sample. No compression wood was evident in any of the samples, nor was the wood unusually resinous.

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The dissected increments were ground in a Wiley mill to wood meal passing a 40-mesh screen and screened on a 60-mesh sieve in order to retain only the 40 to 60-mesh fraction. The meal was allowed to air dry in the laboratory, and the moisture content was determined by ovendrying a portion of each sample. The ovendried material was discarded after weighing, since the high temperature required may adversely affect the yield of cellulose produced.

Table I. Characteristics of Trees Sampled

Site	*Crown class*	*Tree No.*	*D.b.h. in.*
Good	Dominant	1	26.5
		2	30.6
	Intermediate	3	11.4
		4	11.5
Poor	Dominant	5	11.7
		6	10.1
	Intermediate	7	5.7
		8	5.0

Duplicate 2-g. portions from each of the samples of wood meal were submitted first to ether and then to alcohol-benzene extractions. Both of the extractions were carried out in a Soxhlet apparatus for a 6-hr. period. Cellulose was determined by the Cross and Bevan method, following essentially the procedure outlined in TAPPI Standard T 17 m-46. Yields were expressed as a percentage of the calculated ovendry weights of the initial material.

Summerwood percentage was also determined for each of the sapwood and heartwood samples, since some workers have demonstrated a relation between summerwood percentage and cellulose yield (*2*). Summerwood was defined as those cells in which the double cell wall was thicker than the lumen, as determined on radial microtome sections. This definition generally coincided with the dark, dense summerwood bands readily visible with the naked eye in the outer portion of each growth ring.

RESULTS AND DISCUSSION

Analysis of Eight Trees

The results of the Cross and Bevan cellulose determinations on eight trees are shown in Table II. The individual yields reported are the averages of two replicates whose standard deviation was 0.64%. If trees no. 4 and no. 5 are momentarily discounted, it can be seen that the cellulose yields from the remaining six trees fall within rather narrow limits from 60.4 to 62.4%.

Analysis of variance revealed that site, crown class, radial position, and their interactions were all insignificant factors in regulating cellulose yields. Growth rate expressed as average ring width likewise failed to explain variation in cellulose content among trees, since the regression of percentage cellulose on growth rate was not significant. Similarly, summerwood percentage gave an insignificant regression, although values

Table II. Cross and Bevan Cellulose Yields for Eight Douglas-Fir Trees

Tree No.	Cellulose, per cent of ovendry weight: 16th–25th rings	5 outermost sapwood rings	Average
1	60.8	60.0	60.4
2	63.0	61.8	62.4
3	62.3	60.0	61.2
4	54.3	55.5	54.9
5	65.9	64.8	65.4
6	62.1	60.7	61.4
7	61.6	62.4	62.0
8	60.4	61.0	60.7

for summerwood ranged from 21 to 51%. Since per cent summerwood and specific gravity of wood are closely related in Douglas-fir (*3*), specific gravity also would have doubtful merit in predicting cellulose yields between trees. Zobel and McElwee (*1*) also found no consistent relationship between specific gravity and cellulose yields among loblolly pines. However, it is generally accepted that the proportion of cellulose in summerwood is higher than in springwood. This is probably true within a single tree, and in fact may account for the increase in cellulose content usually observed from pith to bark, but its influence may be overshadowed by other factors when making comparisons between trees at comparable ages.

Although the main factors contributed little to the variation in cellulose content, an independent test showed the difference between the extreme trees (no. 4 and 5) to be highly significant. Furthermore, a very real difference exists between trees 3 and 4, which represent replicate individuals of the same crown class growing on the same site. This also has been demonstrated in loblolly pine by Zobel, *et al.* (*4*), who conclude that variation in cellulose among trees of the same age growing on the same site is always considerable. In order to confirm the rather wide variation observed in cellulose between trees no. 4 and no. 5, water-resistant carbohydrate (WRC) and alpha cellulose were determined in these trees by using a modified version of the chlorite technique of Wise, *et al.* (*5*). This modification was developed by Yundt and Bradway† for the use of Zobel and McElwee in evaluating loblolly pine (*1*). The results of duplicate determinations are summarized in Table III. The numerical values are naturally different from those of the Cross and Bevan technique, but a statistically significant difference still exists between trees at the 1% level. Thus the results reaffirm the superiority of tree no. 4 over tree no. 5. The method of Yundt and Bradway can be applied on 0.75-g. samples of wood meal in a fraction of the time necessary for the classical Cross and Bevan procedure, and therefore it is to be preferred in future assessment of cellulose content.

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Table III. Yields of Water-Resistant Carbohydrate and Alpha Cellulose from Extreme Trees

Tree No.	Determination, per cent of ovendry weight	16th–25th rings	5 outermost sapwood rings	Average
4	WRC	70.0	72.9	71.4
	Alpha cellulose	43.1	48.1	45.6
5	WRC	77.7	76.2	77.0
	Alpha cellulose	51.7	51.5	51.6

Table IV. Cross and Bevan Cellulose Yields as Related to Age from Pith (Tree No. 2)

Age, yr.	Cellulose, per cent of ovendry weight
1–5	57.4
6–15	58.9
16–25	61.6
26–35	61.2
36–45	60.2
46–55	61.4
56–65	63.4
66–75	62.9
76–81	63.3

Since the difference between trees cannot be ascribed to any of the growth or environmental factors studied, it must be concluded that the difference is due to some factor unaccounted for in the present experiment. It is entirely within reason to speculate that the variation between trees may be an inherent one under genetic control. Such was the conclusion reached by Schütt (*6*) when studying the variation in cellulose content in lodgepole pine. In his study, greater differences were found among various provenances of the pine growing under the same site and climatic conditions than could be found between sites.

Analysis of a Single Tree from Pith to Bark

The results of the Cross and Bevan cellulose determinations on tree no. 2 are presented in Table IV. Values shown are the means of duplicate determinations. The two lowest cellulose yields correspond to those samples representing rings 1 to 5 and 6 to 15. In succeeding years, cellulose content fluctuates, but nevertheless shows a tendency to increase with age at a much slower rate than for the first 15 yr. The results are therefore comparable with those of Zobel, *et al.* (*4*), who have consistently found lower amounts of cellulose in the core (juvenile) wood than in the outer wood of the southern pines.

CONCLUSIONS

Based on the analysis of eight Douglas-fir trees, no fully satisfactory explanation for the variation in cellulose yields could be found. However, it is reasonable to suggest that most of the variation in cellulose content could be attributed to inherent genetic characteristics of the individual trees.

The work of Zobel and McElwee (*1*) and the results of this study indicate that certain steps might be taken to improve the quality of pulpwood. The degree of between-tree variation in cellulose content suggests the advantage of selecting trees with high cellulose yields for breeding purposes. However, this objective must be reached without neglecting other factors of wood quality such as growth rate, specific gravity, and fiber length. Similarly, features of the living trees such as self-pruning ability, branch size and resistance to decay should not be overlooked. In such a selection program, an index of prospective pulp yield combining growth rate, specific gravity, and cellulose content, should perhaps be used as a leading criterion of wood quality.

In the preceding experiment, the range of Cross and Bevan cellulose was 10.5% (54.9 to 65.4). Such trees as those representing the lower limit of this range would be undesirable from breeding, even if their other wood quality features were better than average. On the

other hand, the tree displaying the highest cellulose yield would also be undesirable if its other internal characteristics were unsatisfactory. It might be more justifiable to select one of the trees providing an intermediate yield of cellulose, if corresponding growth rate, specific gravity, and fiber length values were superior. Progeny tests must be conducted, however, to determine the degree of hereditability of these wood quality factors before the development of high-yield strains can be said to be entirely feasible. Furthermore, it would be desirable to conduct pilot pulping tests on samples removed from trees deemed superior on the basis of the internal characteristics noted above. This would make it evident whether the increase in yield and quality of the pulp produced would warrant the necessary expenditure on a tree-improvement program.

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Degradation of Pectic Acid by Some Delignifying and Bleaching Agents

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Methods for preparing apple pectic acid of 97% purity and of fairly high viscosity in 1% NH_4OH were briefly noted. The rates of decrease in viscosity caused by conditions simulating those used in preparing wood holocelluloses suggested that pectic acid was degraded less by the chlorination than by the chlorine dioxide and the sodium chlorite procedures. The lower viscosity D.P. often observed for chlorite holocelluloses might therefore reflect the behavior of polyuronide constituents rather than that of the cellulose itself. Sodium hypochlorite and hydrogen peroxide at 25°C. and pH 8 to 12 degraded pectic acid rapidly. Galacturonic acid was prominent, and DL-tartaric acid was present, among the degradation products from all five oxidants; no other products were detected by chromatographic methods after chlorination or after bleaching with hydrogen peroxide. Chlorine dioxide, acidified sodium chlorite and alkaline hypochlorite also produced mucic acid, but chlorine dioxide differed from the other two oxidants in its failure to yield tartronic acid, and in its comparative indifference to the pH.

Almost thirty years ago, Schmidt and his collaborators (*1*) showed that chlorine dioxide in aqueous pyridine slowly removed the lignin from wood meals and left the "skelettsubstanz" or holocellulose in a fibrous, insoluble state. Jayme and Hanke (*2*) replaced the chlorine dioxide with acidulated sodium chlorite, and this modification was rendered more convenient by Wise and co-workers (*3*, *4*). In its present form, the method employs aqueous sodium chlorite at not more than 80°C. and in the pH range 3.5 to 6.5. The other principal laboratory-scale method of delignification, originated by Ritter and Kurth (*5*, *6*, *7*) in 1933, consisted of alternating brief chlorinations of the moist woodmeal at 0 or 20°C. with extractions by hot alcohol-pyridine or alcohol-ethanolamine. General considerations would suggest that the local concentration of by-product hydrogen chloride from the chlorination, although transient, would be intense enough to cause some degradation of the carbohydrate portion. Staudinger and Jurisch (*8*) found that this expectation was justified in the case of cotton linters, which were degraded much more rapidly by chlorine water at 20°C. than by aqueous chlorine dioxide. Comparisons of the degradative effects of chlorination and of aqueous sodium chlorite in the preparation of holocelluloses from woodmeals, however, for years gave conflicting results. Timell and Jahn (*9*), whose article included a detailed review of the relevant literature, then showed conclusively that chlorinations reduced the viscosity degree of polymerization (D.P.) of cotton linters more than did aqueous sodium chlorite, but the effect of these two agents when used for the preparation of paper birch holocellulose was reversed. Timell's subsequent (*10*) investigation of jackpine gave the same result. It seemed possible that the cause of this anomaly lay in the behavior, not of the cellulose proper, but of a constituent that was relatively resistant toward acid hydrolysis, and was susceptible to oxidative degradation by chlorite or chlorine dioxide. Polyuronosides, such as pectic materials (I) or uronoside residues in hemicelluloses, at once came under consideration.

The well-known resistance of pectins toward acid hydrolysis was recently discussed by Whistler and Richards (*11*) who showed that a glycoside of 4-*O*-methyl-D-glucuronic acid was hydrolyzed about 20 times more slowly than was the glycoside of the corresponding sugar. On the other hand (*12*), galacturonic acid (II) appeared to be oxidized to mucic (III), DL-tartaric (IVa and b) or tartronic acid (V) more readily by aqueous chlorine dioxide or chlorite than by chlorine water in the conditions used for delignifying woods, and the easy oxidation of galacturonic acid to

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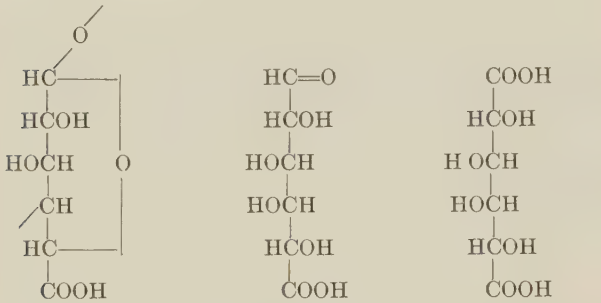
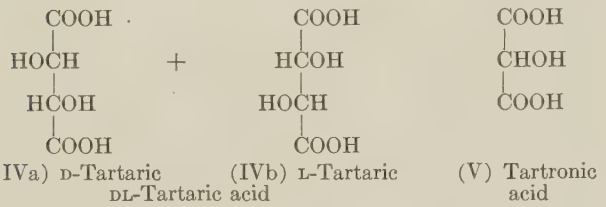
the same products with alkaline hypochlorite was also noted. Golova and Mayat (*13*) observed that methyl-α-D-galacturonoside was more readily oxidized than methyl-α-D-glucoside by acidic sodium periodate and by basic hydrogen peroxide systems. Golova and his collaborators (*13, 14*) also showed that the viscosity of aqueous sodium polygalacturonate from oranges decreased rapidly in the presence of oxygen or hydrogen peroxide near 95°C., but not at 20°. Earlier oxidations were reviewed in the book by Kertesz (*15*). The present research concerned the behavior of high visocisity pectic acid toward the delignifying agents already mentioned, and also toward the bleaching effect of alkaline hypochlorite and alkaline hydrogen peroxide.





Pectic acid was isolated from a commercial apple concentrate by various combinations of published methods (*16*), some of which were detailed in the experimental portion. The most advantageous combination found (Table I) consisted of precipitating crude pectic acid methyl ester (crude pectin) by diluting the concentrate with acidified aqueous methanol. The conversion of pectin to pectic acid by means of acids or enzymes never appeared to have been satisfactory, and resort was made to deësterification with alkali, in spite of the degradation involved (*17, 18*). Trial showed that the mildest conditions yielding a nearly methoxyl-free product involved saponification with 1% NaOH near 20°C. for about 90 min., or were more drastic than those described in some previous methods. It was also possible without decreasing purity to saponify the original concentrate and to omit the isolation of the crude pectin.

The addition of calcium chloride to the saponified liquor precipitated calcium pectate, which was decomposed to insoluble pectic acid by 1% hydrochloric acid. A part of the product was reprecipitated as calcium pectate and recovered, but the uronic acid content was not greatly increased and the intrinsic viscosity was reduced by about 10% (Table I). Exceptionally thorough washing of the first calcium pectate precipitate, and also of the regenerated pectic acid, with water, gave an equivalent purification without decrease in viscosity. The most convenient final purification (Stage 4) consisted of a selective autohydrolysis to remove residual araban and leave a product which after complete hydrolysis yielded hardly discernible traces of nonacidic sugars on paper chromatograms. This pectic acid, a dusty, light-brown powder, representing about two-thirds of the pectin in the original concentrate, was used in subsequent experiments.

Table I. Isolation and Purification of Pectic Acid

Stage	*Uronic Anhydride, %*	$[\eta]^a$
1. Crude pectin	67	..
2. Saponification,[b] recovery as Ca salt, then as pectic acid[c]	87	3.25
3. Reprecipitated as Ca salt, recovered as pectic acid[c]	(89)	(2.99)
4. Pectic acid after autohydrolysis[d]	97.6	2.8
5. Pectic acid methyl ester[e]	..	1.5[e]
6. Methyl ester, nitrated[f]	..	0.34[f]
7. Methyl ester saponified,[b] recovered as pectic acid[c] (yield 50%)	..	1.4

[a] Intrinsic viscosity in 1% NH_4OH.
[b] With 1% NaOH near 20°C. for 80 to 90 min.
[c] Calcium or sodium ions removed at pH 1.5 or less.
[d] At pH 2 and 60°C. for 24 hr. OCH_3, 0.3%.
[e] Methylated with diazomethane to OCH_3, 17.4% (calc. for methyl pectate, OCH_3, 16.3%). Viscosity in water.
[f] With "nondegrading" HNO_3-H_3PO_4-P_2O_5 mixture. Viscosity in acetone.

Although the intrinsic viscosity of nitrated pectin could be determined in acetone (*19, 20*), the nitration of the above pectic acid with a nitric acid-phosphoric acid-phosphorous pentoxide mixture thought to be nondegrading toward cellulose (*21*) yielded insoluble products. Attempts were therefore made to convert the pectic acid to its methyl ester in several ways, but Vollmert's methylation with diazomethane (*22*) was the only one to yield a water-soluble product with nearly the correct methoxyl content. The corresponding nitrate was soluble in acetone, but its intrinsic viscosity, $[\eta]$, of only 0.34 suggested that degradation had been extensive; another pectic acid, of $[\eta]$, 0.75 in 1% NH_4OH, also yielded a nitropectin of $[\eta]$, 0.25 to 0.30. Most of this degradation occurred during the nitration (Table I, Stages 5 and 6), although the change in solvent from water to acetone rendered an exact comparison impossible. This result was contrary to expectation, because the nitration of native pectin with red, fuming nitric acid appeared to cause little degradation in previous cases (*19, 20*). The degradation of approximately 50% that occurred during the methylation with diazomethane agreed with the recent observation of Neukom and Deuel (*17*) and was at variance with earlier opinions (*22, 23*). Alcohol as well as carboxyl functions were substituted by the diazomethane, because about one fourth of the methoxyl groups in the methyl pectate resisted saponification. Vollmert (*22*) found diazomethane to be highly selective for the acidic groups in his pectic acid. As expected, the methyl pectate was degraded by saponification, the recovery of insoluble pectic acid being only 50% (Table I, Stage 7).

Attempts to estimate the D.P. of pectic acid from the intrinsic viscosity of the nitropectin were abandoned at this point in favour of determinations made with solutions of pectic acid in 1% aq. NH_4OH. No reliable correlation was available between the intrinsic viscosity

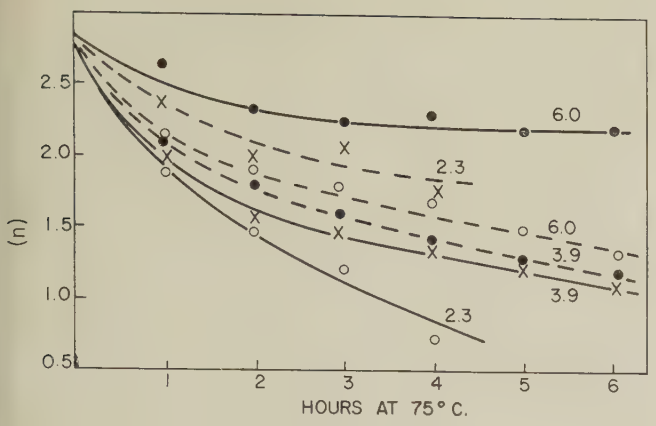


Fig. 1. Plot of intrinsic viscosity of pectic acid with 10-fold molar amounts of oxidant at pH values shown on plots. Solid lines, 0.14 M sodium chlorite; broken lines, 0.14 M chlorine dioxide

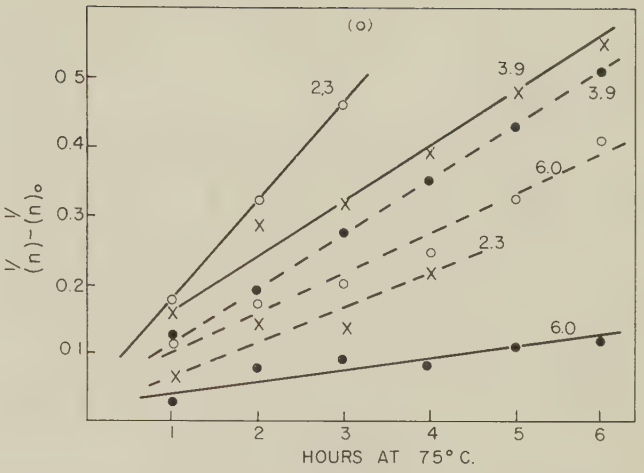


Fig. 2. Rate plots of increase in intrinsic fluidity of pectic acid as in Fig. 1 at pH values shown on plots. Solid lines, 0.14 M sodium chlorite; broken lines, 0.14 M chlorine dioxide

and the D.P., but data published by Schmidt (*24*) for the osmotic pressure and intrinsic viscosity of related systems suggested that the relationship D.P. = 100 $[\eta]$ might be valid. On this basis, a D.P. of about 300 was attributed to the pectic acid used in the present work.

The pectic acid was degraded at 70 to 75°C. by immersion in a 10-fold base-molar amount of 0.14 *M* ClO_2 or sodium chlorite buffered to within 0.2 of a pH unit. The chlorine dioxide formed as a by-product in the oxidations with sodium chlorite was continuously expelled by the passage of nitrogen gas preheated to 70°C. and the observed effects were accordingly attributed to the chlorite (*25*). After various times, the bleached products were recovered by filtration in yields that ranged from 80 to almost 100% by weight, and their intrinsic viscosities were determined in 1% NH_4OH. As expected from previous work (*26*), the degradation caused by chlorine dioxide was almost independent of the acidity (Fig. 1, broken lines), while the action of sodium chlorite increased greatly as the acidity increased from pH 6 to pH 2.3. Chlorine dioxide and sodium chlorite were thus independent oxidants, each with its characteristic behavior, and their degrading action on pectic acid was similar only near pH 3.9. Although a 70% reduction in the relative molar amount of chlorite caused a slight decrease in the rate of degradation, the addition, after four hours, of more chlorite, or of chlorine dioxide, to the respective systems described in Fig. 1 (pH 3.9) did not affect the later shapes of the plots. The results were therefore not seriously affected by the exhaustion or decomposition of the oxidants.

When the data of Fig. 1 were re-plotted as increase in intrinsic fluidity against time, all the plots approximated to straight lines passing through the origin (Fig. 2). This behavior was characteristic of a uniform linear polymer whose monomeric units were all equally exposed to the chance of degradation (*27*). There was thus no evidence that the pectic acid contained units degraded with particular ease, or that its failure to dissolve had rendered a portion inaccessible to the oxidants. Pallmann and Deuel (*28*) observed practically no degradation of methyl pectate or sodium pectate by 0.033 *M* ClO_2 at pH 1 to 6.5 and temperatures up to 90°C., but calculation showed that their samples were of low intrinsic viscosity (probably about 0.12) and probably were highly degraded initially.

In order to simulate the chlorination of wood meal, the water-wet pectic acid was exposed near 0°C. for 5 min. to a stream of chlorine gas. After thorough washing, the insoluble product was submitted three times more to chlorination, portions being withdrawn after each cycle for examination. The overall recovery of pectic acid was 90%; the product was lighter in color than the original pectic acid, but was not white. Since a plot of the intrinsic fluidity against time was linear (Fig. 3, lower plot), the moist pectic acid was presumably completely accessible during chlorination. The rate of degradation during chlorination near 0°C. was similar to that observed with sodium chlorite at pH 6 and 75°C. (Figs. 1 and 2), but was clearly much less than those pertaining to chlorite or chlorine dioxide in the more acid systems used in the delignification of woods. Although a pectic acid chlorinated for 5 min. was not visibly altered by extraction with hot monoethanolamine-ethanol or pyridine-ethanol, the amine salts so formed were partly soluble in water. After recovery, the intrinsic viscosities of both the soluble and insoluble products were only about three quarters of the initial value. The overall degradation in the chlorination-extraction cycle might thus amount to 35%, or be

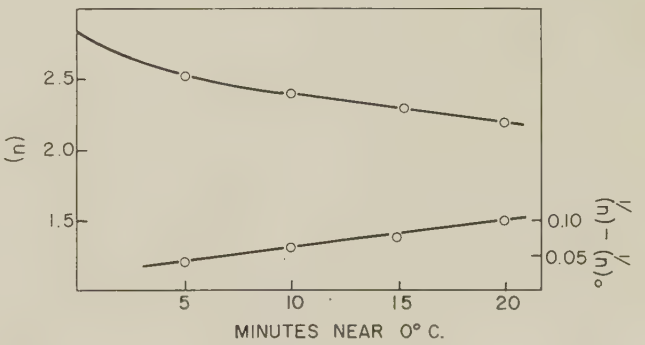


Fig. 3. Degradation of water-wet pectic acid by repeated 5-min. treatments with chlorine gas. Upper plot and left-hand ordinates, decrease in intrinsic viscosity; lower plot and right-hand ordinates, increase in intrinsic fluidity

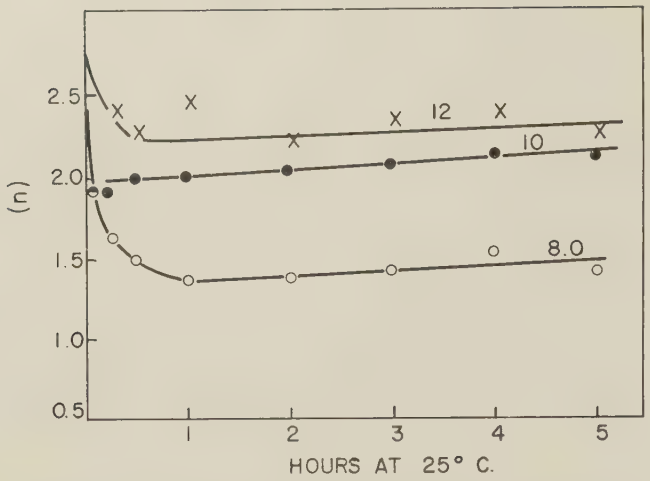


Fig. 4. Change in intrinsic viscosity of pectic acid in 3-fold molar amounts of 0.06 M sodium hypochlorite at pH values noted on plots

comparable to that observed in the chlorite treatment. In the preparation of holocellulose from wood, the chlorination, but not the extraction, step caused degradation (*9*).

The addition of a 10-fold molar amount of calcium hypochlorite at room temperature to pectic acid caused the immediate formation of insoluble calcium pectate, but when the mixture was acidified no pectic acid was precipitated. This excessive degradation led to the use of a 3-fold molar amount of sodium hypochlorite, but the recoveries of the bleached, insoluble pectic acids were still only 60 to 85%. The hypochlorite caused a very rapid initial degradation of the pectic acid (Fig. 4), and a slow, small increase in intrinsic viscosity then occurred. This increase was perhaps caused by the accumulation of carboxyl groups in the oxidized pectic acid, and almost certainly signalized the breakdown of a direct relationship between intrinsic viscosity in 1% NH_4OH and the D.P. The plots, however, did show in a qualitative fashion that degradation was much more extensive at pH 8 than at pH 10 or 12, a result already well known for the degradation of cellulose by hypochlorite (*29*).

In agreement with the work of Deuel (*30*), pectic acid was very rapidly degraded by hydrogen peroxide and the yields of bleached products ranged from 14 to only 70% in experiments made at 25°C. for 1 or 2 hr. at pH 3, 8, and 12. The corresponding viscosities were erratic. An attempt to follow the degradation with peroxide of pectic acid homogeneously dissolved in 1% NH_4OH ended in failure because the gas evolved interfered with the determination of the viscosity. This gas contained carbon dioxide which presumably originated in a decarboxylation of uronic acid groups. According to Deuel (*30*), the action of hydrogen peroxide on pectin was greatly dependent on the presence of catalysts and anticatalysts, and this circumstance might explain the divergence of the present results from the negative outcome of the experiments made at room temperature by Golova and his collaborators (*13*).

A series of small-scale oxidations was carried out in order to study the nature of the products by paper partition chromatography. The recovered pectic acid on hydrolysis yielded galacturonic acid, but also faint traces of a series of sugar acids having R_f values between 0.51 and 0.67 in the solvent system used (butanol–formic acid–water). These sugar acids were probably oxidation products of the carbohydrate associated with the pectic acid, whose purity was about 97%. No further study was made of the pectic acid recovered, but attention was diverted to the residual mother liquors. After the removal of excess oxidant and of cations, concentration of the liquors often caused the separation of crystalline mucic acid; the filtrate from the mucic acid then yielded chromatographic evidence of some or all of the following substances: D-galacturonic acid, DL-tartaric acid, and tartronic acid. Two larger-scale oxidations, the first with aqueous sodium hypochlorite in excess at pH 12 and the second with excess sodium chlorite at pH 2.5, made it possible to isolate each of the above three substances in a crystalline condition and to prove their identity beyond doubt.

As shown in Table II, galacturonic acid was prominent among the products from all five oxidations, and in this respect the behavior of pectic acid was similar to that of methyl-α-D-galacturonoside (*12*). The assumption that galacturonic acid (II) was the primary product led at once to the inference that mucic acid (III) was next formed and yielded DL-tartaric acid (IV) by symmetrical cleavage at the 2,3 and 4,5-positions. Sodium chlorite and hypochlorite also oxidized mucic acid in the 3,4-position to give 2 moles of tartronic acid (V), but chlorine dioxide, as previously observed (*12*), did not have this capacity.

Table II. Principal Oxidation Products of Pectic Acid[a]

Oxidant	*Mucic acid*	*Galacturonic acid, R_f 0.04*	*Tartaric acid, R_f 0.21*	*Tartronic acid, R_f 0.38–44*
Chlorine dioxide[b]	+	++	+	0
Sodium chlorite	+	++	+	+
Chlorine gas	0	++	+	0
Sodium hypochlorite	++	++	+	+++
Hydrogen peroxide	0	++	+	0

[a] Oxidations lasted 90 or 150 min. A relatively large or intense spot on paper chromatogram denoted by +++; a fair amount by ++; a trace by +. Paper chromatograms in butanol–formic acid–water system.

[b] A trace of unknown sugar acid, R_F 0.12, also present.

Previous articles (*31–34*) referred to the presence of up to 3% of pectic materials in various woods, and to the possibility that these materials, and also the uronic constituents of the hemicelluloses, might be trans-esterified. Since pectins readily formed gels of very high viscosity, the contribution of the polyuronides to the intrinsic viscosity of the holocellulose or its nitrate might originally be substantial. The present results suggested, although they did not prove, that this contribution would be decreased only slightly by chlorination of the wood, but that some of the degraded uronic material would be removed in the subsequent extractions with alcoholic amine and with water. On the other hand, treatment of the wood with chlorine dioxide or sodium chlorite would degrade the polyuronosides more severely; the degraded materials would for the most part remain in the holocellulose, and lower its observed viscosity.

EXPERIMENTAL

Preparations of pectin and pectic acid, after precipitation from solution and washing, were solvent-exchanged through anhydrous methanol into acetone and were then dried *in vacuo* over phosphorous pentoxide at room temperature. The products appeared to be free of adsorbed methanol (*35*), because the methoxyl contents observed for several pectic acids solvent-exchanged and dried in this way were less than 0.5%. All solutions were concentrated in a rotary film evaporator at not more than 50°C. All analyses were reported, on an ash and moisture-free basis, as the mean of concordant duplicates. Incidental solvents such as methanol were carefully purified and dried.

Materials and Analytical Methods

A commercial concentrate of "Certo" apple pectin, known to be of recent manufacture, served as the source of the pectic acid. The sample was very viscous, had pH 3.1, gave a positive iodine test for starch and yielded a superior gel when added to a 66% solution of sucrose. Found: total solids, 5.1%.

To determine moisture and ash, 1-g. samples were dried at 105°C. for 3 to 4 hr., and after being weighed the residue was heated near 630°C. in a muffle furnace. On a micro-scale, 0.01- to 0.03-g. samples were ashed as described by Niederl and Niederl (*36*). Uronic acids and furfural were determined by distilling samples with 12% HCl according to the Browning (*37*) and the TAPPI Standards, respectively. It was assumed that the uronic anhydride present yielded 21.5% of its weight as furfural, and the observed yield was decreased by this amount before calculating the pentosan content. The content of pectin (or of partly esterified pectic acid) was accepted as the sum of the uronic anhydride and $^{17}/_{31}$ of the methoxyl content. Hexosans were found by subtracting the sum of the pectin and pentosan percentages from 100%.

Pectic substances were prepared for chromatography by hydrolyzing 1-g. samples, first for 1 hr. at 25°C. in 9 ml. of 72% H_2SO_4 and then, after dilution with 250 ml. of water, at 15 p.s.i. steam pressure for exactly 1 hr. (*38*). The acid was removed as barium sulphate and the filtrate, now approximately 1 liter in volume, was de-cationized by Amberlite IR-120 ion-exchange resin. After cautious evaporation in stages to 3 to 5 ml., the volume was brought up to 10 ml. before aliquots of 10 λ (10^{-2} ml.) were withdrawn for paper partition chromatography. Details about the latter procedure, in which butanol–formic acid–water was the solvent and bromphenol blue was the spray, were reported in the previous article (*12*). D-Galacturonic acid, DL-tartaric acid, and tartronic acid were readily distinguished and separated because their R_F values were 0.04, 0.21, and 0.38 to 0.44, respectively. The solvent systems used for the identification of nonacid sugars were *n*-butanol–pyridine–water (10:3:3 vol.), which failed to separate mannose from arabinose, and ethyl acetate–acetic acid–water, which did not separate glucose and galactose. The previous article (*12*) also described larger-scale separations on a Dowex 1-4X anion-exchange column (acetate form) by elution with N acetic and 0.5 *N* trifluoroacetic acid.

To determine the intrinsic viscosity of pectic acid, a 0.05-g. sample, corrected for ash and moisture, was dissolved in 10 ml. of 1% NH_4OH. Solution was usually complete within 1 or 2 hr., but any trace of insoluble material was removed on the centrifuge. An Ostwald viscometer kept at 30°C. was used; the solution was then diluted with its own volume of solvent and the new specific viscosity determined, the process being repeated until high dilution made the results unreliable. A graphical extrapolation of the quotient $\eta_{sp./c}$ to zero concentration then gave the intrinsic viscosity $[\eta]$ %. Schmidt (*24*) correlated osmotic pressure measurements with the intrinsic viscosity of a 50% esterified methyl pectate dissolved in 0.1 *N* HCl or in 0.05 *M* sodium citrate and established a relationship equivalent to D.P. = 100 $[\eta]$ %. The equation for nitrated pectin in acetone was equivalent to D.P. = 170 $[\eta]$ %.

Isolation of Crude Pectin

The "Certo" concentrate was diluted with an equal volume of water and was acidified to pH 0.5 with dilute hydrochloric acid. Technical methanol was then added to 66% concentration, and the pH was maintained at 0.7 to 0.9 by the addition of more acid. It was essential that the pH be not greater than 1.5 if the precipitated pectin was to have a low ash content; at pH 1.9, 2.5, and 3.0, the ash contents were 4.4, 7.2, and 9.0%, respectively. The precipitated gel was recovered on the centrifuge, was washed with technical methanol until free of chloride ion, was solvent-exchanged through anhydrous methanol into acetone, and dried. Found: uronic anhydride, 66.8; furfural, 20.7; OCH_3, 8.1; pentosans, 6.4; hexosans, 23.4; ash, 2.0%. An experiment on a much smaller scale, with an older commercial apple pectin concentrate of much lower viscosity, yielded a product of markedly different composition. Found: uronic anhydride, 81.8; furfural, 18.6; OCH_3, 9.2; pentosans, 0.9; hexosans, 12.3; ash 0.24%.

The light-brown powders from both preparations readily formed clear solutions in water, 1% NH_4OH or ammonium oxalate, but were insoluble at room temperature in aqueous sodium hydroxide of more than 1.3% concentration. Although lack of solubility in sodium hydroxide was sometimes caused by the presence of calcium ion (*39*), the low ash content of the samples and their ready solubility in water suggested that in the present case some other factor was operative. When hydrolyzed and chromatographed on paper, the crude pectins gave intense spots for galacturonic acid, and large to moderately large spots for glucose, galactose, and arabinose; traces of mannose, xylose, and rhamnose were also present.

Isolation of Pectic Acid

Preliminary experiments showed that an aqueous solution of the above, low viscosity, pectin was incompletely saponified after a few minutes at pH 8.5 (*40*), or when this pH was maintained until it ceased to change (*41*). Solution for 30 min. in 100 parts of 2% NH_4OH was also ineffective (*42*), but 1.33% aq. NaOH within 1 hr. at room temperature yielded a pectic acid with OCH_3, 0.2%. Reduction of the concentration to 1% caused an increase to 0.5% in the methoxyl content of the product. The procedure

reach completion. The liquor was then prepared for chromatography as in previous cases (Table II). Hydrogen peroxide in equimolar amount was also added to a homogeneous solution of pectic acid in 1% NH_4OH at pH 8, but a gas was evolved which interfered with the determination of viscosity. This gas caused a white precipitate to appear in lime water, and presumably contained carbon dioxide.

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Corrugating Pulp from Wheat Straw by Cold Soda Process

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Cold soda pulping was appraised for its applicability to the preparation of corrugating pulps from wheat straw. Whole and chopped straws were steeped for 1, 3, and 5 hr. with 6 to 16% NaOH based on straw. Various vacuum and pressure impregnation treatments were investigated. A 3-roll sugarcane press was used to expel steep liquor from the straw and to fiberize steeped straw. High yields, 75 to 84%, of pulps were obtained with chemical consumption of 6.2 to 7.5% from initial concentrations of 12, 14, and 16% NaOH. Pulps were converted on a 10-in. laboratory fourdrinier machine to 9-point corrugating board having high crush resistance. Reuse of steep liquor through 12 complete pulping cycles was accomplished without appreciable change in the physical properties of the pulps. Since high-quality pulps are attainable by cold soda pulping, possibly future investigation of continuous processes could reduce the high (9:1) liquid-solid ratios employed in these laboratory studies and lower the initial chemical concentration required.

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In recent years straw pulping has declined in the United States. Fluctuating availability of sound wheat straw in certain areas is a partial cause, although more efficient processes are also needed by the strawboard industry. Actually, any continued use of wheat straw requires a constant search for improved handling and pulping operations which will produce a satisfactory product at a competitive price. Techniques in pulping that would reduce operational costs would be beneficial to producers of straw corrugating medium. As part of a long-range program on straw pulping at the Northern Division the cold soda process that proved applicable to hard woods was tested, and the results are the basis of the present report.

Cold soda pulping was developed at the Forest Products Laboratory (*1*, *2*) to process hardwoods abundantly available in the United States, particularly in the south. This process has been used in both batch and continuous operations; the resultant pulps are suitable for the manufacture of corrugating board, printing papers, and tissue. The success of this

pulping technique has been attributed to the high yield of pulps having adequate properties.

Some interest in pulping straw without heating has been reported as early as 1930. At that time Wells (*3*) claimed the development of a straw pulping technique that involved steeping in cold caustic soda followed by defibering and refining in a rod mill. A few years later this was followed by Skul'skava's report (*4*) that maceration of straw with cold caustic soda required 3 days and that the resulting experimental papers were deficient in strength properties. The patent granted to Marsoni (*5*) in 1941 describes the preparation of cellulose pulps by macerating several straws in caustic soda solutions of 3 to 4° Bé. for 6 to 12 days at room temperature. Milk of lime was used in the studies of Bertrand and Delga (*6*) who steeped straw for 4 days at 40°C. to prepare papermaking pulps. Conspicuous in these reports are the long steeping periods. A shorter steeping period, 16 hr., was used by Francais (*7*) but the chemical requirement was 24% NaOH based on dry weight of straw. Recovery of unused chemicals from steep liquor for reuse was a feature of a patent claim of Skolnik (*8*) who used a 2% solution of caustic soda in pulping straw; however, temperatures used were just short of boiling.

characteristics such as burst, tensile, and tear were also determined on the board.

EXPERIMENTAL

A wheat straw (Kawvale) from central Illinois was used either whole or chopped to 1- to 3-in. lengths. The straw was steeped in an unheated 1.6 cu. ft. stainless-steel digester tumbling at 1 r.p.m., using a liquid-to-solids ratio of 9:1. Vacuum and/or pneumatic pressure was applied to the digester in experiments that included these process modifications. Caustic soda concentrations of 6 to 16%, based on the dry weight of straw (approximately 0.7 to 1.8% solution) were used with steeping periods of 1 to 5 hr. Two procedures were followed for handling the straw following the steep:

A. In preliminary runs using a 3- and 5-hr. steeping period the straw was removed from the digester, washed to remove residual liquor, and transferred to a washer-beater to defiberize it.

B. In most of the runs steeped straw was removed from the digester, passed through a three-roll sugarcane press, and transferred to a washer-beater for further defiberizing and washing. The liquor removed by the press was collected, weighed, and analyzed for residual alkali.

Table I. Yields and Strength Properties of Pulps with Variation in Chemical Concentration and Steeping Time

Straw		*Steeping conditions*				*Pulp yields*		*Physical properties of handsheets*				*Physical properties of machine sheets*							
		Chemical, %													*Breaking length, m.*		*Tear factor*		
Exp. no.	*W—Whole C—Chopped*	*Initial*	*Used*	*Pulp pressed*	*Time, min.*	*Crude, %*	*Washed, %*	*Burst factor*	*Breaking length, m.*	*Tear factor*	*Concora B.W.*	*Freeness of run, S.-R., ml.*	*Density, g./cc.*	*Burst factor*	*M.D.*	*C.D.*	*M.D.*	*C.D.*	*Concora B.W*
1	C	8	..	No	180	87.2	81.6	19	4320	54	52	585	0.44	14	4465	2898	43	51	46
2	C	10	..	No	180	80.1	76.2	25	4940	50	49	610	0.56	20	5510	3660	39	51	63
3	C	12	..	No	180	77.0	74.1	30	5700	50	71	610	0.63	26	6940	4280	38	53	76
4	C	14	..	No	180	74.7	71.5	28	5600	50	74	585	0.63	25	6600	4280	38	50	73
5	W	12	6.5	Yes	60	83.7	79.0	25	4990	57	61	600	0.48	21	5800	3610	47	53	70
6	W	14	7.4	Yes	60	82.4	78.5	25	4750	57	56	605	0.52	25	6560	4040	43	55	82
7	W	16	7.5	Yes	60	81.8	77.1	31	5940	48	77	605	0.57	27	7080	4320	38	46	84
8	C	12	6.4	Yes	60	81.4	77.4	26	5080	48	64	600	0.55	24	6220	3940	44	48	78
9	C	14	7.4	Yes	60	78.5	75.7	31	5420	46	71	605	0.67	32	7980	4610	38	50	94
10	W	12	6.7	Yes	180	77.0	73.2	38	5890	55	71	..	..	..	..	..	..	..	..

In view of current knowledge the cold soda process merited further attention to assess its applicability and to devise techniques for its efficient use in the production of high-quality corrugating pulp from wheat straw. Success could possibly not only offer more economical production of strawboard with facilities presently available at strawboard mills, but also indicate economies should new installations be contemplated. These were the two objectives of our investigations. Pulp yields, length of steeping periods, chemical requirements, and pulp quality in preparing the corrugating pulps are summarized. Variables in the evaluation were chemical concentration, steeping time, reuse of liquor, vacuum and pressure impregnation, and the use of whole and chopped straw. The concora value (*9, 10*) of board produced was used as a measure of crush resistance. Other strength

All pulps following the washer-beater treatment were pressed to approximately 20% solids content. Yields were determined on washed pulps and on fines removed in washing; beater evaluation tests were made on washed pulps according to TAPPI Standards T 200 m-45 and T 205 m-53, except that the sheet weight was approximately that of 9-point corrugating medium.

Pulps selected for the preparation of board were refined to approximately 600-ml. freeness (Schopper-Riegler) and screened on a Valley 10-cut screen as previously d scribed (*11*). Appropriate samples of 9-point corrugating board were removed from each machine run for physical tests.

To appraise the value of used steep liquor for further pulping, the solution recovered from the cane press was fortified with caustic soda to the alkali concentration of the original liquor in a series of 12 consecutive steeps.

In other runs the residual chemical was determined and the liquor discarded.

RESULTS AND DISCUSSION

Chemical Requirements, Steeping Time, and Pressing

Initial treatment (Procedure A) of the chopped straw using 14% caustic soda and pulping intervals of 3 and 5 hr. was designed to study the general effectiveness of cold caustic steeping in the preparation of a pulp suitable for corrugating board. Steeping time was similar to that for commercial practice to prepare pulp from straw at elevated temperatures and pressures. Tumbling of the digester plus the high liquid-to-solid ratio of 9:1 insured even distribution of chemical on the straw.

Results obtained on 3-hr. steeps with chopped straw are presented in Table I. Longer steeps gave essentially the same results (data not included) except that about 1% lower yields were obtained. In experiments 1 to 4 using a 3-hr. steep, the crude yields of 74.7 to 87.2% obtained indicated that high pulp recoveries were possible. Concora crush values of 71 to 74 were satisfactory for handsheets from pulps prepared at the 12 and 14% level. Concora values on machine board prepared at the 12 and 14% level also were close, 76 and 73, indicating that the pulps were very similar. Burst and concora values obtained on pulps prepared with 8 and 10% NaOH were lower and indicate that if pulps were prepared with less than 12% chemical, they would not be satisfactory for corrugating board.

Pulps in experiments 5, 6, and 7 were prepared by steeping the straw for 1 hr. in the presence of 12, 14, and 16% chemical and by passing the steeped whole straw through a cane press primarily to expel as much of the spent liquor as possible for reuse and to initiate defiberizing of the straw. Reduction in steeping time from 3 to 1 hr. increases pulp yield and productive capacity of the equipment. Caustic soda consumed is in the range 6.5 to 7.5% showing some increase in chemical usage when the starting ratio is changed from 12 to 16%. Bursting and tensile strengths and concora values are slightly lower for pulps steeped the shorter period at the 12% chemical level. The burst and concora values of the machine board were essentially the same.

A comparison of experiments 8 and 9, using 12 and

Table II. Yields, Chemical Requirements, and Strength Properties of Pulps with Reuse of Liquor, 12% NaOH, and 3-hr. Steep

	Chemical		Chem. anal. of liquor			Pulp yields		Chem. Analysis		Physical properties of handsheets[a]				Physical properties of machine sheets							
Exp. no.	Used, %	Recovered, %	Dry solids, %	Organic matter, %	Ash, %	Crude, %	Washed, %	Ash, %	Lignin, %	Burst factor	Breaking length, m.	Tear factor	Concora B.W.	Freeness of run	Density, g./cc.	Burst factor	Breaking length, m. M.D.	Breaking length, m. C.D.	Tear factor M.D.	Tear factor C.D.	Concora B.W.
10	6.7	4.4	3.5	2.2	1.3	77.0	73.2	4.1	15.0	38	5890	55	71	620	0.73	32	7300	4600	40	45	76
11	6.6	4.4	6.1	4.1	2.0	77.3	72.4	3.6	14.9	33	5890	46	68	610	0.72	30	7540	4600	33	42	72
13	6.6	4.4	8.8	6.3	2.5	79.8	74.2	3.7	15.7	29	5840	44	66	630	0.63	28	6070	3930	42	55	77
15	6.6	4.8	11.0	7.9	3.1	80.4	75.8	3.9	15.3	30	5700	46	67	620	0.66	29	6490	4170	41	55	72
17	6.2	4.5	12.2	8.8	3.4	82.2	77.3	3.9	15.6	28	5230	50	65	630	0.60	27	6210	3890	43	55	67
19	6.5	4.5	13.6	10.0	3.6	82.2	77.2	3.5	15.7	31	5800	43	66	630	0.61	28	6450	4120	41	56	74
21	6.7	4.5	14.5	10.4	4.1	80.8	75.5	3.5	14.9	30	5460	43	66	630	0.63	27	5920	4120	41	52	76

[a] Data at 600 ml. S.-R. freeness.

Table III. Yields, Chemical Requirements, and Strength Properties with Application of Vacuum and Hydrostatic Pressure

Straw	Steeping conditions				Pulp yields		Physical properties of handsheets[a]				Physical properties of machine sheets								
Exp. no.	W—Whole C—Chopped	Chemical, % Initial	Chemical, % Used	Pressure	Time, min.	Crude, %	Washed, %	Burst factor	Breaking length, m.	Tear factor	Concorn B.W.	Freeness of run, S.-R., ml.	Density, g./cc.	Burst factor	Breaking length, m. M.D.	Breaking length, m. C.D.	Tear factor M.D.	Tear factor C.D.	Concora B.W.
22	W	12	7.0	Vacuum-90 lb.	5–55	82.8	78.2	29	5460	48	68	610	0.55	26	6940	3990	34	49	90
23	W	14	8.3	Vacuum-90 lb.	5–55	79.6	76.1	32	5650	45	75	605	0.64	30	7740	4420	42	47	94
24	W	16	7.8	Vacuum-90 lb.	5–55	79.5	76.2	29	5510	46	71	600	0.66	31	7410	4370	33	48	98
25	W	14	8.1	Vac.-press. 2-stage steep	5–25–30	77.0	73.9	31	5840	46	72	600	0.64	30	8310	4460	36	43	101
26	C	12	7.0	90 lb.	60	79.9	76.0	29	5600	48	66	620	0.65	28	7030	4370	48	52	85
27	C	14	8.5	90 lb.	60	77.2	73.5	28	5650	47	75	605	0.63	29	7220	4560	33	56	88
28	C	12	6.8	Vacuum-atmospheric	5–55	79.4	75.4	27	5320	50	63	605	0.65	32	6890	4180	50	53	79
29	C	14	7.7	Vacuum-atmospheric	5–55	79.9	75.9	28	5560	47	65	620	0.65	30	6890	4370	43	53	82
30	C	12	6.7	Vacuum-40 lb.	5–55	80.1	76.6	28	5560	48	71	615	0.63	30	6790	4320	44	54	76
31	C	14	8.0	Vacuum-40 lb.	5–55	78.6	75.3	31	6130	46	74	620	0.67	32	6980	4460	48	50	80
32	C	12	7.0	Vac.-atmos.; 4-cycle	5–10	81.4	78.5	30	5700	47	72	615	0.65	28	6220	4460	46	51	83
33	C	14	7.6	Vac.-atmos.; 4-cycle	5–10	78.4	75.5	30	5890	46	70	610	0.68	30	6700	4610	43	53	80

[a] Data at 600 ml. S.-R. freeness.

14% caustic soda on chopped straw, with experiments 3 and 4 show the combined effect of variations in steeping time and passage of steeped straw through the press. Higher yields of pulp, with essentially the same physical characteristics, were obtained by using the press in conjunction with a 1-hr. steep instead of a 3-hr. steep without the press. Slightly higher yields were obtained on the whole straw and higher crush values were obtained on chopped straw under the same conditions of time, chemical concentration, and processing. Examination of the machine board showed that less shive and coarse material were present in board from the chopped straw. Initial freeness of pulps from the whole and chopped straw was higher (800 to 840 ml.) than normally obtained on pulps prepared at elevated temperatures and pressures.

In general, boards had smoother surfaces when prepared from pulps in which longer steeps were used, due to more complete defiberizing.

Reuse of Liquor

Experiments 1 through 9 indicate that chemical concentration of 12 to 16% and a steeping period of 1 to 3 hr. constitute satisfactory conditions for producing a good quality corrugating pulp. The initial amount of chemical in the steep liquor is considerably greater than that consumed in pulping. For the process to be economical it would be necessary to remove liquor from the straw and recover it for subsequent pulping treatments.

To explore the possibility of recovery and reuse of liquor, 12 experiments were carried out on whole straw using 12% NaOH, a 3-hr. steep, and reusing the liquor from each preceding steep. Data are presented in Table II on selected steeps showing chemical consumption of 6.2 to 6.7%. The ratio of liquor-to-straw following pressing was approximately 2:1 with 80% of the original liquor removed for reuse. Pulp yields were in a range of 77.0 to 82.2% with an average yield of 80.0%. Slight increases in pulp yields were indicated following initial treatments. Concora crush values of handsheets were in the range expected on the basis of earlier runs and were not affected by liquor reuse. Analysis of the liquors showed progressive increase in dry solids content (Table II). Although the total ash and organic matter increased in liquors with each succeeding reuse, the increment left by each individual steep decreased progressively. It is possible that in further cycles approximate constancy might be achieved.

Vacuum, Atmospheric, and Pressure Pretreatments

A series of cold caustic treatments was made on the straw to study the effects of vacuum, pressure, or combinations of vacuum-pressure impregnation on yields, chemical consumption, and physical characteristics of pulps prepared under varied conditions (Table III). Pulps were prepared using 12, 14, and 16% NaOH at room temperature, with the application of vacuum followed by application of pressures of 90 p.s.i. for the remainder of the 1-hr. period of steeping. The steeped straw was subjected to mechanical compression in the cane press as described previously. Data indicate a tendency toward slightly higher chemical consumption than for steeps under atmospheric pressure. Yields of pulp were close to those obtained in 1-hr. steeps 5, 6, 7, but concora values on machine sheets were appreciably higher. Utilization of vacuum-pressure technique in preparing pulp from whole straw also improved the surface of the machine board. Lesser amounts of coarse materials were present than in pulps prepared under atmospheric pressure conditions.

Numerous other variations in combinations of pressure and/or vacuum pretreatments are also shown in Table III, but none appeared to have advantage over experiments 22 to 24 just described.

SUMMARY AND CONCLUSIONS

Steeping of wheat straw with caustic soda at room temperature produced pulps with characteristics suitable for the preparation of 9-point corrugating board. With initial concentrations (basis straw) of 12, 14, and 16% NaOH, chemical consumption ranged from 6.2 to 7.5%. Corrugating boards of high crush strength were prepared from pulps obtained in yields of 75 to 84%. Other physical characteristics determined objectively were also in the range of high-quality corrugating media. Good surface characteristics of final boards, as judged by smoothness and the presence of partially defiberized fiber bundles, were favored by use of chopped straw, steeps of 3 hr. in preference to those of 1 hr., a cane press for initial defiberizing and removal of spent liquor from steeped straw, and vacuum-pressure impregnation treatments.

Board of consistently high quality was obtained with pulps prepared by steeping for 3 hr. with spent liquors refortified to 12% alkalinity through 12 complete cycles. Initial freeness values of pulps from cold caustic steeps were generally higher than for those prepared by the customary batch-cooking techniques. This characteristic facilitates washing of chemical from the steeped straw.

Attractive features of the cold soda process for preparing straw pulps are: simplicity of operation, high yields of corrugating pulps, ease of raw material penetration by chemical, reuse of liquor to conserve chemical, and probably adaptability to continuous operation. If the cold soda process is used as a continuous operation, lower liquor-solids ratios and lower initial concentrations of caustic should result.

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Dry Ashing of Pulp and Factors Which Influence It

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It was desired to study the factors which influence dry ash values and the possibility of replacing the conventional ashing method with a sulfation method. It was also of interest to establish whether dry ashing was a satisfactory method of sample preparation for analysis for inorganic contaminants of pulp. Dry ash values were found to decrease with increase in temperature and the decrease varied with the type of pulp. There is a large loss of sodium, calcium, iron, and copper when dry ashing; silica is apparently not lost. A theory is presented to explain this loss. The above results indicate that dry ash data on pulps are of little or no value in determining inorganic contaminants, particularly in comparison between pulps. The technique is also unsatisfactory as a method of sample preparation for sodium, calcium, copper, and iron analyses on pulp. Dry ashing determinations should not be used as an analytical method. Wet ashing should be used exclusively for sample preparation for inorganic analyses except possibly for silica.

Ash values have been widely used throughout the cellulose industries as a measure of the level of inorganic impurities. There are a number of different procedures for ash determinations in the literature and practically speaking, every laboratory seems to have its own conditions for ashing. A number of these methods are summarized in Table I. It is obvious that discrepancies would exist between data taken using such widely different methods. This is further complicated by technique differences such as slight differences in ashing temperature even when the same procedure is used.

The importance of some of the procedure variables which are indicated in Table I would appear to be minor.

Moisture measurements, the desiccant used while samples are being cooled, the sample form, and the calculation method fall into this category. If it is intended that the ash is to be used for iron determinations, hand tearing has been demonstrated to be better than cutting, by direct development of the 1,10-phenanthroline ferrous complex on the sheet. Any cut portion shows definite evidence of iron contamination. The most important variables are probably type of crucibles, burning off and ignition temperature, and time.

Partial attempts have been made to study the effects of these variables by J. E. Jeffery, chairman of the Joint ACS-ASTM Subcommittee on Ash and Inorganic Contaminants and Earl S. McColley, a member of that committee. Their results which are given in the Appendix, indicate that the variables cited appreciably influence the reported ash levels. Apparently all the results in each study were obtained on the same pulp samples. The anions present, which would vary depending on the type wood and the pulping method, might be expected to drastically influence the variation with temperature.

The influence of ashing temperature has been further studied in the recent paper by Bethge and Troëng. (*9*) They have concluded that conventional ashing methods lead to poor reproducibility and reliability and recommend sulfated ash measurements as a standard method for the determination of ash in pulp.

It was therefore desired to study comprehensively the variables in the ashing methods including the influence of types of pulps. This report gives the results of this study along with a study of what contaminants may be lost in the ashing step.

EQUIPMENT AND METHODS

Equipment

Muffle furnaces, multiple electric furnace Type 58, from Elmer and Amend, and two Temco Series 1600 furnaces were used for the study.

Platinum crucibles and dishes of several sizes were used. Colorimetric and flame photometric measurements were made on the Beckman DK and DU spectrophotometers equipped with flame photometer attachments.

All chemicals were "Reagent Grade" supplied by Baker and Adamson.

Methods

Samples of the following types of pulp were obtained:

Type	*Designated in paper*
High D.P., calcium bisulfite base from southern pine	Pulp A
Magnesium base acid bisulfite from spruce and hemlock	Pulp B
Calcium bisulfite base from spruce and hemlock	Pulp C
Prehydrolyzed sulfate from black gum	Pulp D

Alternate sheets (one lot) were used and the other sheets discarded. They were then torn into small squares ($1^1/_2$ to 1 in.) by hand and stored in large bottles. Moistures were run on the samples after 24 hr. and this correction made on all data taken.

The general procedure used for the ash determination is as follows:

Weigh a sample of suitable size (usually 40 g.) into the previously weighed and ignited crucible. Burn off until smoking ceases in a 500°C. muffle with the door closed. Transfer to a muffle of the specified temperature or increase the temperature and maintain for the specified time. No lids are used on the crucible. Remove the crucible and allow to cool in a desiccator containing Drierite. Weigh and calculate the ash.

The sulfated ash was obtained by wetting the ash with concentrated sulfuric acida fter removing from

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Table I. Methods for Ashing Cellulose

Identification of method	*Moisture*	*Sample form*	*Crucible*	*Desiccant*	*Sample weight, g.*	*Burning temp., °C.*	*Burning time*	*Burning conditions*	*Ignition temp., °C.*	*Ignition time*
Ash in wood, TAPPI T 15 m-54	Same sample	Not specified	Pt, alundum, silica, or porcelain	Not specified	2	Not specified	Not specified	Covered	600	1 hr.
Ash in wood, ASTM D 1102–56	Same sample	Ground to pass No. 40 sieve	Pt, silica, or porcelain		2	Not specified	Not specified	Open	580–600	Constant weight
Ash in paper, TAPPI T 413 m-54	Same sample	Not specified	Pt, alundum, silica, or porcelain	Not specified	Not less than 1	Not specified	Not specified	Covered	925	Until carbon eliminated
Ash in pulp, TAPPI T 211 m-54	Same sample	...	Pt, alundum, or porcelain	H_2SO_4 or $CaCl_2$	5	Not specified	Not specified	Open	Not specified	Constant weight
Ash in pulp (*1*), proposed rev. T 211	Separate sample	Specified	Pt, porcelain, or silica	Alumina	Specified	Not specified	Flame ceases	Open with flame	575 ± 25	3 hr. or longer
Ash in wood (*1*), proposed rev. T 15	Separate sample	Specified	Pt, porcelain, or silica	Alumina	5	Not specified	Flame ceases	Open with flame	575 ± 25	3 hr. or longer
Ash in paper (*1*), proposed rev. T 413	Separate sample	Small pieces	Pt, porcelain, or silica	Indicating alumina	Specified	Not specified	Flame ceases	Open with flame	925 ± 25	3 hr. or longer
Laboratory A	Not specified	Torn pieces	Pt	Not specified	20	600	Not specified	Open	800	1 hr.
Laboratory B	Separate sample	Not specified	Porcelain	Not specified	40	500	Smoking ceases	Open	800	1 hr.
Laboratory C	Separate sample	Cut with paper cutter	Porcelain	Indicating Drierite	25	600	Overnight or carbon absent	Open	800	½ hr.
Ash in cellulose acetate, ASTM D 871–54 T	Dry sample	Not specified	Porcelain	Not specified	5, 10, or 50	Flame	Not specified	Open	550–600	3 hr. or longer
Ash in ethyl cellulose, ASTM D 914–50	Dry sample	Not specified	Porcelain	Not specified	2–5	Flame	Not specified	Open	900	1 hr.

Note: All the above procedures call for ignition without a lid.

the muffle after the conventional ashing step, and cooling. The excess acid was removed by heating on an electric hot plate. The sample was then reignited for the specified time.

For the elemental analysis, the ash residue was treated with 5 ml. of concentrated HCl. After evaporating to dryness on a steam bath, the sample was taken up in water, 8 ml. of concentrated sulfuric acid was added, and the sample was diluted to 100 ml. in a volumetric flask. Aliquots were taken for the various analyses.

Silica analyses were made on separate samples which were fused with sodium carbonate after ignition. No analyses for silica were made on the wet ashed samples due to the lack of a satisfactory procedure.

The methods for wet ashing and analyses of elements are the following.

Wet Ashing of Pulps (2). Weigh a sample of approximately 10 g. of air-dried sample and place it into a 1000-ml. Berzelius beaker. Add 40 ml. of 72% H_2SO_4 and break up the pieces of sample with a stirring rod. Place the beaker on a hot plate and heat the sample until the black foaming ceases and the black residue has a solid granular appearance with no liquid visible. Remove from the hot plate and add 30% hydrogen peroxide dropwise until the reaction slows or ceases. Return to the hot plate and evaporate until fumes of SO_3 are given off. If any solid material remains, or the solution is dark, remove and add more peroxide, following by repeating the evaporation step. It may be necessary to repeat this step several times. *Do not add peroxide while the samples are on the hot plate or when SO_3 fumes are being given off.* The acid is now evaporated to approximately 8 ml., adding H_2O_2 occasionally to remove the brown or yellow color. The sample is finally transferred quantitatively to a 100-ml. volumetric flask and diluted to volume.

Iron (3). Standard: 0.1000 g. of pure iron wire is dissolved in dilute HCl and diluted to one liter. The dilute standards are prepared from this concentrated standard.

Procedure: An aliquot of 25 ml. of the sample is pipetted into a 50-ml. volumetric flask. Five milliliters of 10% hydroxylamine sulfate solution are then added and the solution thoroughly mixed. Five milliliters of 40% sodium acetate solution are added to neutralize the excess acid present. The pH should be slightly acid when checked with Fisher Alkacid Tester Paper and if necessary, more sodium acetate solution is added. The red color is developed by adding 5 ml. of 0.1% 1,10-phenanthroline solution. The solution is then diluted to volume and the absorbance at 507 mμ measured with the spectrophotometer. The results are compared with that of a standard iron solution which has been taken through the steps outlined above.

Calculations:

$$\text{p.p.m. Fe} = \frac{\text{micrograms Fe} \times \text{dilution factor}}{\text{ovendry wt. of sample}}$$

Copper (3). Standard: 0.1000 g. of pure copper wire is dissolved in dilute HNO_3, boiled to expel the excess acid, and diluted to one liter. The dilute standards are prepared from this solution.

Procedure: A 25-ml. aliquot of the solution is pipetted into a 125-ml. separatory funnel. The solution is neutralized with concentrated ammonium hydroxide avoiding a large excess of base. Approximately 1 g. of ammonium citrate is added and thoroughly mixed. Five milliliters of 0.1% sodium diethyldithiocarbamate are added and mixed. The solution is extracted three times with 5 ml. carbon tetrachloride collecting the extracts in a 50-ml. volumetric flask. Add 1 ml. methanol to clear the solution and dilute to volume. The absorbance is determined at 440 mμ on a spectrophotometer and compared with standards carried through the above procedure.

Calculations:

$$\text{p.p.m. Cu} = \frac{\text{micrograms Cu} \times \text{dilution factor}}{\text{ovendry wt. of sample}}$$

Flame Photometric Method for Calcium (2). Standard: 5.0464 g. of $CaCO_3$ are dissolved in a small amount

of dilute HCl and then diluted to 2 liters. Aliquots of this are measured into 500-ml. volumeteric flasks, 30 ml. of concentrated H_2SO_4 are added, and the samples diluted to volume.

Procedure: A portion of the solution obtained from the ashing step is placed in the 5-ml. beaker provided with the flame photometer. The spectrophotometer switch is turned to *Check* and allowed to warm up for a few minutes. The flame is lit and adjusted. With the wavelength set at 422.7 mμ, the switch at 0.1, the shutter open, and the transmission knob at 0, distilled water is introduced into the flame. The dark current knob is adjusted so that the galvanometer reads 0. A standard containing 100 p.p.m. calcium is now introduced into the flame and the sensitivity knob adjusted until the galvanometer reads 0 with the transmission knob set at 100. Other standards are introduced into the flame and the transmission knob rotated until the galvanometer reads 0. The readings are plotted on linear graph paper to give a standard curve. The unknown is now introduced into the flame and the transmission knob rotated as before to obtain the reading. Two or more readings approaching the zero setting from each direction should be taken and the readings averaged. The concentration of the unknown may now be obtained from the standard curve.

Calculations:

$$\text{p.p.m. Ca} = \frac{\text{micrograms Ca in solution of unknown} \times \text{dilution factor}}{\text{ovendry wt. of sample}}$$

Flame Photometric Method for Sodium (4). Standard: A 0.1 *N* NaOH solution is carefully standardized against potassium acid phthalate (NBS). Aliquots of this NaOH solution, carefully neutralized with 0.1*N* HCl to phenolphthalein and diluted to a specific volume with a solution containing 8 ml. concentrated sulfuric acid per 100 ml. are used as standards for the sodium curve.

Procedure: A portion of the solution obtained from the ashing step is placed in the 5-ml. beaker supplied with the flame photometer. The spectrophotometer switch is turned to *Check* and allowed to warm up for a few minutes. The flame is lit and adjusted. With the wavelength set at 589 mμ, the switch at 0.1, the shutter open, and the transmission knob at 0, distilled water is introduced into the flame. The dark current knob is adjusted so that the galvanometer reads 0. A standard containing 100 p.p.m. Na is now introduced into the flame and the sensitivity knob adjusted until the galvanometer reads 0 with the transmission knob set at 100. Other standards are introduced into the flame and the transmission knob rotated until the galvanometer reads 0. The readings are plotted on linear graph paper to give a standard curve. The unknown is now introduced into the flame and the transmission knob rotated as before to obtain the reading. Two or more readings approaching the zero setting from each direction should be taken and the readings averaged. The concentration of the unknown may now be obtained from the standard curve.

Calculations:

$$\text{p.p.m. Na} = \frac{\text{micrograms Na} \times \text{dilution factor}}{\text{ovendry wt. of sample}}$$

Silica (3). Standard: 0.100 g. of pure SiO_2 is fused with sodium carbonate in a platinum crucible. The sample is dissolved in a small amount of dilute HCl and then made basic with NaOH. This concentrated standard is stored in a polyethylene bottle. Dilute standards are prepared by diluting this standard with water.

Reagents: Ammonium molybdate solution—Dissolve 75 g. of ammonium molybdate in water containing 322 ml. of 1:9 sulfuric acid. Dilute to 1000 ml.

Reducing agent: Dissolve 7 g. anhydrous sodium sulfite in 100 ml. of water. Add 1.5 g. 1,2,4-aminonaphthol sulfonic acid and after dissolving, add a solution of 90 g. of sodium bisulfite in 800 ml. of water. Dilute to 1000 ml. 10% tartaric acid solution.

Procedure: Add 2 g. of anhydrous sodium carbonate to the residue in the crucible. Fuse by heating in a muffle at 900°C. Remove and gently swirl the crucible to spread a thin layer of the molten material over the bottom and lower sides of the dish. Cool and dissolve the residue in 10 ml. of water. Carefully neutralize with dilute HCl and transfer to a 50-ml. volumetric flask. Dilute to volume and take an aliquot equivalent to approximately 2 g. of the original sample and transfer to another 50-ml. volumetric flask. Add 5 ml. of ammonium molybdate solution and mix. After 5 min. add 5 ml. of 10% tartaric acid solution and then 5 ml. of the reducing solution. Dilute to volume and after 5 min. read at 820 mμ. Compare with standards carried through the same procedure.

Calculations:

$$\text{p.p.m. } SiO_2 = \frac{\text{micrograms } SiO_2 \times \text{dilution factor}}{\text{ovendry wt. of sample}}$$

RESULTS AND DISCUSSIONS

Preliminary Experiments

Burning Off Conditions. The proposed revisions of TAPPI Standard T 211 m-54 (ash in pulp), T 15 m-54 (ash in wood), and T 413 m-54 (ash in paper) permit the sample to burn with a flame. It has been pointed out that this practice results in a mechanical loss of the ash (*5, 6, 7*).

To further check this point, ashes were run after burning off the sample as specified in these proposed procedures with a final ignition at 660°C. followed by sulfation. This is compared in Table II with data taken on the same pulp using the same procedure except that the muffle door was closed and no flames were evident. This is taken as a positive indication that loss occurs when a flame is permitted.

This loss may be attributed either to a mechanical loss or to the fact that the sample is actually at a higher temperature due to the presence of the flame (*9*). In the latter case, the loss may be due to volatilization of the salts.

Preliminary Investigation of Ashing Step. In order to establish what happened during the ashing step, a study was made using pulps A, B, and C. Both porcelain and platinum crucibles were used and the ashing time and temperatures were varied. Iron, and, to a limited extent, chlorine measurements were made, since it was felt that these elements might be readily lost at the higher temperatures. The results of this study are compiled in Table III. Duplicate analyses of each

Table II. Pulp A

Sample treatment	*Sulfated ash, %*	
Sample ignited with flame	0.256	0.245
	0.247	0.241
	0.244	0.244
	Av. 0.246; std. deviation = 0.0053	
Muffle door closed (no flame)	0.276	0.279
	0.267	0.276
	0.275	0.290
	Av. 0.277; std. deviation = 0.0076	

condition were run and the results averaged.

The porcelain crucibles used for the pulp A ashes were badly attacked in all cases and the ash in all porcelain crucibles ignited at 950°C. was found fused with the crucible itself.

From these data, it was noted that in general, ash levels decreased with both increases in temperature and time. The demonstrated loss of iron and chlorine or conversion of all anions to oxides was insufficient to account for the changes observed. The influence of temperature apparently varied with the pulp also.

To establish these relationships more firmly, a statistically designed experiment was undertaken to study the effects of time and temperature on the results of the dry ash and the sulfated ash methods.

Since there appeared to be a variation in effect between the pulps, pulp D was added to the samples. Due to the reaction of the ash with the porcelain crucibles, platinum dishes were used for all the determinations.

Statistically Designed Experiments

The experiments were carried out in the form of a nine-point Boxonian design (*8*); the arrangement being shown in Fig. 1. Each circle in the figure shows a combination of time and temperature at which duplicate measurements were taken, for each of the four pulps and for the dry ash and sulfated ash methods. The

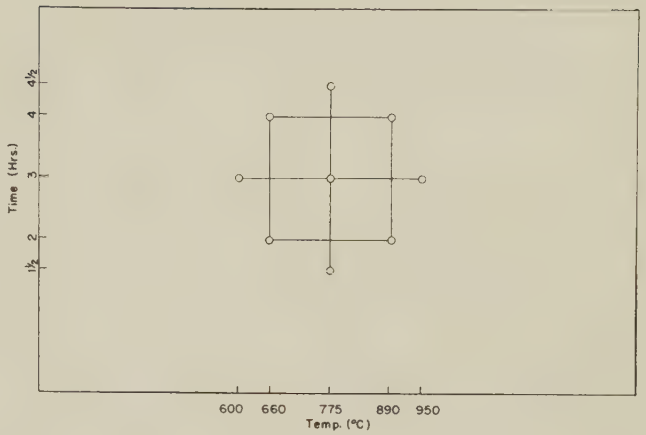

Fig. 1. Experimental design used for each of the four pulps in complete random order

center point was repeated and all ten experiments were run in a completely randomized order.

The sides of the square in Fig. 1 are two units long, while the wing points are 1.5 units from the center point. A unit was 115°C. for temperature and 1 hr. for time. The ten treatment combinations (including the duplicated center point) are given in Table IV along with the observations for the four pulps and two methods. The chronological order of running the experiments is indicated by the numerals in column 1.

The results were analyzed by standard regression methods in which response surfaces were fitted for the four pulps and two methods separately. The nature of the equation that was fitted, in a least-squares sense, was full second order equation, i.e., it included second powers and the cross-product of the variables. The duplicate measurements were used to give an estimate of the error of the method and to test the goodness-of-fit. The errors were assumed to be homogeneous for each set of results.

Table III

Pulp	*Ashing temp., °C.*	*Ashing time, hr.*	*Porcelain crucibles*			*Pt crucibles*	
			Ash, %	*Fe, p.p.m.*	*Cl, p.p.m*	*Ash, %*	*Fe, p.p.m*
A	600	3	0.205	7.8	95	...	...
	600	4	0.205	5.5	50	...	...
	600	5	0.195	...	35	...	...
	800	1½	0.160	8.4	5	...	...
	800	2	0.150	2.8	0	0.11	10.4
	800	3	0.135	4.6	..	...	...
	950	1	0.095	9.8[a]	0	0.06	6.3
	950	2	0.11	7.3[a]	0	0.06	10.4
	950	3	0.105	3.7[a]	..	0.05	4.8
B	600	3	0.125	...	0	...	...
	600	4	0.110	3.3	..	...	...
	600	5	0.110	2.4	..	...	...
	800	1½	0.110	2.2	0	...	...
	800	2	0.105	2.0	0	...	...
	800	3	0.115	2.2	0	...	...
	950	1	0.060	5.6[a]	..	0.04	3.2
	950	2	0.095	5.2[a]	..	0.03	11.1
	950	3	0.100	5.3[a]	..	0.07	7.0
C	600	3	0.130	1.7	65	...	...
	600	4	0.130	2.3	50	...	...
	600	5	0.125	1.7	..	...	...
	800	1	0.120	1.8	..	...	...
	800	2	0.095	2.1	..	0.05	7.3
	800	3	0.110	0.5	30	...	...
	950	1	0.055	6.5[a]	..	0.04	4.4
	950	2	0.095	4.9[a]	0	0.06	4.4
	950	3	0.09	3.5[a]	..	0.04	5.8

[a] Crucibles badly attacked, part of iron may be from crucibles.

Table IV

Chronological order	*Temp., °C.*	*Time, hr.*	*Pulp*	*Ash, %*	*Sulfated ash, %*
10	600	3	A	0.222, 0.217	0.295, 0.293
			B	0.092, 0.091	0.140, 0.136
			C	0.144, 0.149	0.210, 0.212
			D	0.043, 0.045	0.084, 0.082
9	660	2	A	0.211, 0.213	0.273, 0.285
			B	0.096, 0.095	0.139, 0.138
			C	0.149, 0.135	0.208, 0.191
			D	0.035, 0.036	0.081, 0.077
6	660	4	A	0.216, 0.212	0.260, 0.257
			B	0.106, 0.091	0.132, 0.112
			C	0.147, 0.125	0.200, 0.183
			D	0.051, 0.044	0.067, 0.066
4	775	$1^1/_2$	A	0.168, 0.175	0.228, 0.239
			B	0.083, 0.077	0.120, 0.116
			C	0.107, 0.084	0.161, 0.141
			D	0.035, 0.033	0.074, 0.072
1	775	3	A	0.122, 0.157	0.173, 0.215
			B	0.073, 0.078	0.105, 0.118
			C	0.060, 0.096	0.104, 0.154
			D	0.034, 0.045	0.059, 0.077
8	775	3	A	0.175, 0.146	0.234, 0.199
			B	0.061, 0.075	0.087, 0.102
			C	0.109, 0.130	0.173, 0.199
			D	0.036, 0.038	0.069, 0.076
2	775	$4^1/_2$	A	0.179, 0.151	0.240, 0.195
			B	0.051, 0.071	0.073, 0.096
			C	0.081, 0.114	0.127, 0.163
			D	0.044, 0.046	0.072, 0.074
5	890	2	A	0.092, 0.088	0.102, 0.098
			B	0.044, 0.045	0.053, 0.053
			C	0.072, 0.071	0.112, 0.116
			D	0.034, 0.035	0.060, 0.064
7	890	4	A	0.099, 0.083	0.127, 0.108
			B	0.049, 0.026	0.067, 0.035
			C	0.066, 0.051	0.096, 0.071
			D	0.045, 0.036	0.071, 0.063
3	950	3	A	0.048, 0.062	0.037, 0.052
			B	0.029, 0.034	0.029, 0.033
			C	0.053, 0.054	0.073, 0.072
			D	0.042, 0.041	0.061, 0.059

The results of this analysis are interpreted to mean that there was no detectable effect of time on the ash values, either alone or in interaction with the temperature.

The relation between ash values and temperature was different for each of the four pulps and also for the two methods. These relationships are shown graphically in Figs. 2 to 5. Generally speaking, the ash values decreased with increasing temperature. Further, the sulfate ash values were usually higher than the dry ash results but the curve has a steeper slope.

The residual values, that is, the deviations of the observed values from the fitted curves, were examined for indications of any systematic time trend. There were none. The spread of these residuals was compared with the variation of duplicate measurements to see if the variability unaccounted for by the regression curves was similar to the variability expected from the lack of reproducibility of the analytical procedure and sample variation. The two were similar in every case. This is shown in Table V, where the standard deviations of the residuals are given alongside the standard deviation of the results estimated from duplicate observations. These are for single observations.

A comparison of Table IV with the graphs in Figs. 2 to 5 indicates the precision varies with the general level of the measured ash values; but it may be a difference in variability between the pulps.

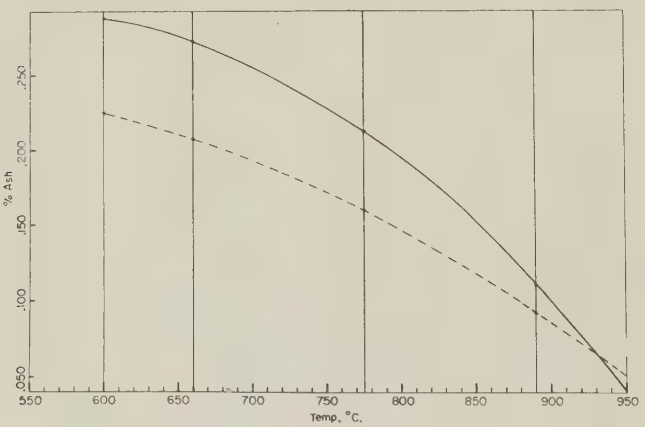

Fig. 2. Pulp A

– – – –Dry ash method. ——Sulfate ash method.

Loss of Metal Ions in Dry Ashing

In order to establish what was being lost as a function of temperature in the statistical study, analyses were made of the metal ion concentration after ashing under various circumstances. These were wet ashing, conventional ashing at 660°C., ashing at 660°C with sulfation, final ignition at 890°C., and conventional ashing at 890°C. All dry ashing was done in platinum dishes. The wet ashing method was the sulfuric acid peroxide procedure.

The results of triplicate analyses were averaged and are summarized in Table VI. Assuming that there is little or no loss in the wet ashing procedure, the data indicate that there is more loss of the metal ion in almost every other procedure.

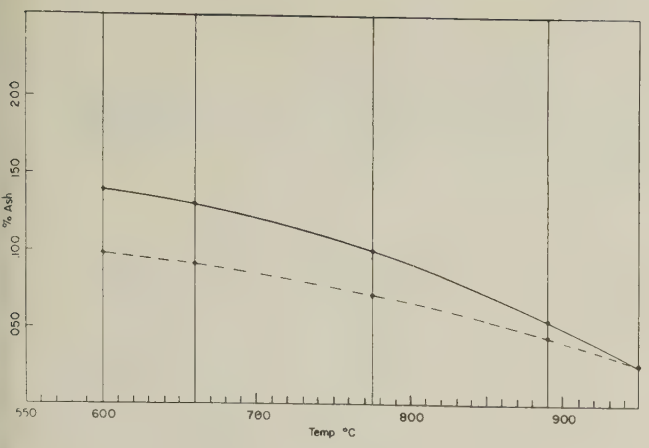

Fig. 3. Pulp B

– – – –Dry ash method. ——Sulfate ash method.

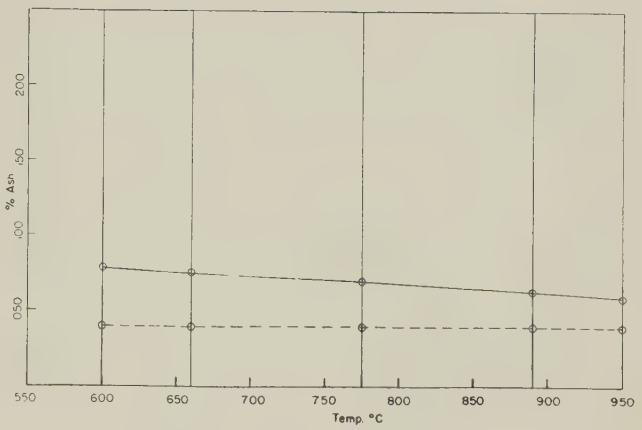

Fig. 5. Pulp D

– – – –Dry ash. ——Sulfate.

The observed loss of sodium is of particular interest since except for pulp D, sodium salts make up the major portion of the inorganic contaminants. It is this loss which is shown in the higher temperature ashing and it may be speculated that the low sodium value is the reason for the relatively low sensitivity to

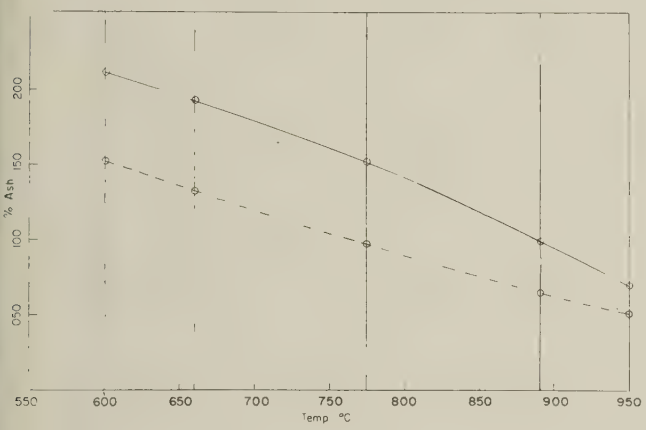

Fig. 4. Pulp C

– – – –Dry ash method. ——Sulfate ash method.

ashing temperature exhibited by pulp D. Since no reported sodium salt of an inorganic acid boils or sublimes even near the lower ashing temperatures used (660°C.), it is hard to understand how this loss can occur. It can be postulated that the metal ion is present in part as the salt of carboxylic acid end groups on the cellulose. On degradation it would be possible that volatile low molecular weight organic salts such as sodium acetate, sodium formate, etc., are being produced.

This theory is not necessarily refuted by the data on the sulfated ashes since the organic salts could presumably be lost along with the excess sulfuric acid. The fact that the values for the sulfated ash are higher than those which have been conventionally ashed at the same temperature (890°) indicates that the sulfates are at least not as volatile as the salt form which exists in the conventional ashing.

Table V. Standard Deviations in Per Cent Ash

Pulp	Dry ash		Sulfated ash	
	Residuals	Duplicates	Residuals	Duplicates
A	0.013	0.013	0.017	0.017
B	0.009	0.008	0.013	0.011
C	0.015	0.015	0.020	0.017
D	0.005	0.004	0.006	0.005

The loss of calcium parallels that of sodium. It is apparent that the loss is not so drastic and the relatively level values for dry ashed samples suggest that some portion of the calcium is present in the original pulp in a form which is potentially volatile.

Iron is lost during dry ashing probably through volatilization of the chloride salt. This is indicated by higher losses where appreciable amounts of chloride are present such as in the case of pulp A. In the case of this pulp sufficient chloride is present to cause an inter-

Table VI

Pulp	Ashing conditions	Ca, p.p.m.	Na, p.p.m.	Fe, p.p.m.	Cu, p.p.m.	SiO_2, p.p.m.
A	WA	54	669	4.5	8.8	...
	660	37	577	3.8	4.3	96.8
	SO_4	36	494	2.9	1.0	90.4
	890	34	321	2.3	1.9	96.4
B	WA	51	272	3.6	3.1	...
	660	56	235	3.7	2.2	44.0
	SO_4	34	165	1.9	1.1	28.1
	890	42	46	1.9	1.6	53.0
C	WA	169	493	2.4	5.6	...
	660	139	382	0.7	2.4	43.8
	SO_4	136	381	0.0	2.0	45.1
	890	149	286	0.7	0.9	41.3
D	WA	207	45	3.5	5.9	...
	660	126	15	1.0	4.1	88.5
	SO_4	72	20	0.0	0.9	85.0
	890	92	16	0.6	1.9	80.8

WA—wet ash; SO_4—sulfated dry ash.

ference in the 1,10-phenanthroline method due to the attack on the platinum dishes. Therefore the iron data on the dry ashed samples of pulp A were taken on samples ashed in porcelain crucibles.

Copper shows the same loss pattern as the other metal ions studied.

Silica analyses were not run on the wet ashed samples because of possible difficulties from the glass reaction beaker. The relative consistency of the silicas on the dry ashed samples seems to indicate that ashing temperature has very little effect on the silica. No explanation is available for the inconsistencies in pulp B. It seems indicated that dry ashing is at least partially satisfactory for sample preparation for silica determination.

CONCLUSIONS

Dry ashing of pulps gives ash data which are a function of the temperature of ashing with the effect of temperature varying depending on the composition of the ash (or the type of pulp). This implies that comparative dry ash data between pulps have no meaning. When dealing with one type of pulp where the salt composition is constant and the ratio of these salts is constant, dry ash data may be of value provided the temperature is rigidly controlled. In certain types of pulps, the slope of the ash temperature curve is so steep that it is doubtful that the temperature can be controlled adequately in conventional muffles.

There is a loss of sodium, calcium, iron, and copper in most pulps regardless of the dry ashing conditions. Silica is apparently not lost in most cases in dry ashing. The mechanism of this loss may be associated with the fact that these ions may be present at least in part as the salts of carboxylate ions in the cellulose. On degradation it is possible that volatile salts of low molecular weight organic acids are formed. There are also indications that chloride ion concentrations may influence the loss of certain ions such as iron.

RECOMMENDATIONS

It is recommended that dry ash measurements be eliminated as a control procedure except under special circumstances.

Since alkali and alkaline earth cations make up the largest portion of inorganic contaminants in pulp and are readily determined using wet ashing and a flame photometer, it is recommended that these analyses replace the ash figure. It is further recommended that wet ashing be used as a method of sample preparation when sodium, calcium, iron, and copper analyses are to be run.

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It is also desired to thank Dr. J. E. Jeffery and Dr. Earl S. McColley for permission to use the data given with Appendix.

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APPENDIX

Data from Earl S. McColley

Determination of Ash of Cellulose

20-g. samples conditioned at 42% R.H., 78°C.

Time heated over burner, min.	*Heated in muffle — Temp., °C.*	*Heated in muffle — Time, hr.*	*Ash, %*
	Coors Crucibles No. 3		
10–20	600	2 1/2	0.042
		3 1/2	0.042
		1 1/2	0.043
		4 1/2	0.037
	800	1 1/2	0.037
		2	0.041
30–40	600	2 1/2	0.041
		3 1/2	0.040
		4	0.037
		1	0.051
		4	0.043
	800	4	0.043
		3	0.033
50–60	600	1	0.034
		2 1/2	0.038
		3 1/2	0.037
		1	0.034
	Vitreosil Dishes, 100 mm.		
5–10	600	2	0.071
		1	0.074
		4	0.042
	800	1	0.059
		2	0.052
		4	0.039
20–30	600	3	0.054
		2	0.047
		1	0.041
		2	0.076
	800	1	0.041
		2	0.039
		3	0.049
60–90	600	3	0.050
		4	0.042
	800	3	00.44
		4	0.051
	Platinum Dishes, 75 mm.		
4–15	600	1	0.048
		2	0.039
	800	1	0.037
		2	0.040
		2 1/2	0.038
		2 1/2	0.040
20–40	600	1	0.036
		1 1/2	0.036
		2	0.035
		2 1/2	0.041
	800	1	0.036
		2	0.040

Data from J. E. Jeffery

Ignition temp., °C.	*Ash, %*
500	0.165
550	0.165
600	0.150
650	0.130
700	0.105
750	0.105
800	0.105
850	0.100
900	0.100
950	0.095

The Use of Polysulphides in the Kraft Pulping Process

J. R. PECKHAM and M. N. MAY

A mixture of southern pinewoods was pulped by the kraft process, using conditions considered to be typical of industrial practice, to produce a bleachable grade of pulp. For comparison purposes, the same supply of wood was pulped to a similar permanganate number level using an alkaline liquor which was compounded to make it rich in polysulphides. Triplicate digestions were made in each case. The polysulphide pulp was found to have a screened yield advantage over the kraft pulp of up to 2%, based on moisture-free wood. The pulp color and bleachability compared favorably with that of the kraft pulp. The pulp quality was somewhat inferior in the case of the polysulphide pulp. Consideration of the pulping data led to the conclusion that commercial application of the polysulphide pulping system would be dependent upon the relationship between the value of the pulp which was obtained in excess over that which could be realized from conventional kraft pulping, and the additional costs imposed by the modified recovery system. Accelerated digester corrosion would probably not be a deterrent in the use of the soda-sulphur system.

RENE Berthiér (*1*) has reported significant increases in pulp yields when maritime pine (*Pinus pinaster*) was cooked with an alkaline liquor rich in polysulphides in comparison with similar digestions using conventional kraft liquor. This study was undertaken to establish whether similar results could be obtained in pulping a mixture of southern pines.

RAW MATERIALS

Approximately equal parts of three species of southern pines (longleaf, loblolly, and slash) were blended for the wood supply. Solutions of technical grade sodium hydroxide and purified crystals of sodium sulphide were used for the kraft liquors. Flowers of sulphur added to the sodium hydroxide solution provided the polysulphides in the so-called "soda-sulphur" cooks.

EXPERIMENTAL

The attack on this problem involved performing triplicate digestions using typical kraft pulping conditions and comparing the results with similar digestions when a sodium hydroxide-elemental sulphur liquor was substituted for the kraft liquor. The conditions used and the product variables obtained are shown in Table I.

The soda-sulphur liquor was prepared by dissolving 1200 g. of flowers of sulphur in 24.6 l. of a caustic solution which contained 305.6 g. per l. NaOH. The mixture was heated to 50°C. and stirred occasionally. The resulting solution was analyzed for total sulphur, sodium thiosulphate, sodium sulphite, sodium sulphide, and sodium sulphate according to the methods of Kesler (*2*). Sodium polysulphides were determined by the method of Berthiér (*1*). These results are summarized in Table I.

Sulphidity for the soda-sulphur cooks is defined as the ratio of "active" sulphur to active alkali, both calculated as sodium oxide. The "active" sulphur is considered to be the difference between the total sulphur analysis and the amounts of sodium thiosulphate and sodium sulphate present in the liquor and includes polysulphide sulphur. The calculation may be expressed:

$$\text{Sulphidity} = \frac{\text{total S (as } Na_2O) - (Na_2S_2O_3 + Na_2SO_4, \text{ as } Na_2O)}{\text{active alkali (as } Na_2O)}$$

Total and active alkali were determined by TAPPI Standard T 624 m-44. The normal kraft liquor, also analyzed for total and active alkali by the above method, was prepared by mixing solutions of sodium hydroxide and sodium sulphide of known concentrations in such a manner that the sulphidity was approximately 30%. Sulphidity for the kraft cooks was defined as the ratio of sodium sulphide to active alkali (both as Na_2O) in the usual manner.

The digester used in this work was a stainless steel vessel of 44-l. capacity, equipped for external heating and forced circulation. The chips were charged to the digester along with the desired amounts of chemical and water, and the cover was secured. At the end of the cooking cycle, the internal pressure was relieved to 80 p.s.i. and the contents of the digester were discharged through a cyclone separator. The pulp, collected in a muslin-covered box, was transferred to a stainless steel wash tank equipped with a perforated false bottom. In washing, three dilutions were made using approximately 65 gal. of warm water in each. The first dilution was stirred for 10 min. and the second for 5, using a Patterson Uni-power stirrer. The third dilution was not stirred. All effluent from the wash tank was directed into a muslin-covered washbox, and entrained fibers were returned to the pulp mass. Screening was carried out on a small Valley flat screen fitted with 0.010-in. slotted plates. The rejected portion of the pulp was ovendried, weighed, and discarded. The weight appears as "screenings" in the yield figures and is reported as a percentage of the unscreened pulp.

The accepted pulp was caught in a tank identical with the wash tank and was partially dewatered by draining the tank. The effluent from the tank was treated as described for the washing process to minimize loss of fines. The partially dewatered stock was transferred to a laundry-type centrifuge which was lined with a coarse screen. The first effluent from the centrifuge was passed through the pulp cake to retain fines. The cake of pulp removed from the centrifuge was broken into bits in a mechanical device designed for this purpose, and the crumbs were packaged in poly-

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Table I. Cooking Conditions and Product Variables

Polysulphide cooking experiments

Type of cook→	Cook 1 (Soda-sulphur)	Cook 2 (Soda-sulphur)	Cook 3 (Soda-sulphur)	Av. (Soda-sulphur)	Cook 4 (Kraft)	Cook 5 (Kraft)	Cook 6 (Kraft)	Av. (Kraft)
Sulphidity, %[a]	30.0	30.0	30.0	30.0	28.8	28.8	28.8	28.8
Permanganate no.	23.7	24.3	24.7	24.2	26.3	24.2	23.2	24.6
Yield, %								
Unscreened[b]	50.3	50.3	50.7	50.4	48.6	48.7	48.7	48.7
Screened, %[b]	50.0	50.0	50.2	50.1	48.2	48.1	48.0	48.1
Screenings[c]	0.46	0.56	0.94	0.65	0.88	1.31	1.38	1.19

Constant Conditions

Chip charge (moisture-free basis), g.	5638	Time to maximum temperature, min.	105
Water ratio, cc./g.	4.0	Time at maximum temperature, min.	90
Active alkali (as Na_2O), %[b]	17.0	Blowdown to 80 p.s.i., min.	5
Maximum temperature, °C.	170		

Analysis of Soda-Sulphur Liquor

Total alkali, as Na_2O, g./l.	221	Sodium sulphide, as S, g./l.	11.86
Active alkali, as Na_2O, g./l.	220	Polysulphide, as S, g./l.	14.05
Sodium thiosulphate, as S, g./l.	13.25	Sodium sulphate, as S, g./l.	1.85
Sodium sulphite, as S, g./l.	0.45		

Analysis of Kraft Liquor

Total alkali, as Na_2O, g./l.	170.5	Sulphide, as Na_2O, g./l.	49.1
Active alkali, as Na_2O, g./l.	170.5		

Note: Soda-sulphur liquors analyzed using methods of Berthiér (*1*) and Kesler (*2*). Kraft liquor analyzed using TAPPI Standard T 624 m-44. Sulphidity was determined iodometrically.

[a] For the soda-sulphur cooks, sulphidity was defined as: $\frac{\text{total S} - (Na_2S_2O_3 + Na_2SO_4)}{\text{active alkali}}$.

[b] Basis moisture-free wood.

[c] Basis moisture-free unscreened pulp.

ethylene bags. Representative samples removed at this time were weighed, ovendried at 105°C., and reweighed to establish the moisture content of the pulp. From these data the yield of pulp, based on moisture-free wood, was determined.

The unbleached pulps were beater evaluated, using TAPPI Standards T 200 m-45, T 205 m-53, and T 220 m-53. The results are shown in Table II.

The bleaching characteristics of the six pulps were investigated by subjecting them to a three-stage bleaching sequence. This was done by applying the same amount of chlorine to each pulp in a chlorination stage, extracting with sodium hydroxide, and applying a bleachability test to the chlorinated and extracted pulps. The data concerning these experiments appear in Table III.

DISCUSSION OF RESULTS

The digestions made with sodium hydroxide and sulphur (cooks 1–3) were specially designed to have a high polysulphide content as compared with the regular kraft cooks 4 to 6. The permanganate number for the soda-sulphur pulps averaged 24.2 and for the kraft cooks, 24.6. At this permanganate number, an average unscreened yield of 50.4% was obtained for soda-sulphur pulps and 48.6% for kraft pulps. Screening rejects were lowest for the soda-sulphur pulps, 0.65 as compared to 1.19% for the kraft pulps.

The pulps were essentially the same color and behaved similarly when bleached. The soda-sulphur pulps consumed a total of 10.9% chlorine to achieve a brightness of 80% G.E. The average for the kraft pulps was 10.7.

The pulp strength data shown in Table II were plotted against beating time. From these graphs, the pulp properties at 700 and 400-ml. Schopper-Riegler freeness were interpolated. Table IV shows the results obtained when the various physical characteristics were compared at the two freeness levels. Figure 1 compares the two kinds of pulps when the tearing strengths are plotted against tensile strengths. From these data it would appear that the kraft pulps are somewhat superior to the soda-sulphur pulps, especially in bursting and tensile strengths and folding endurance. The differences are more striking at the 700-ml. freeness

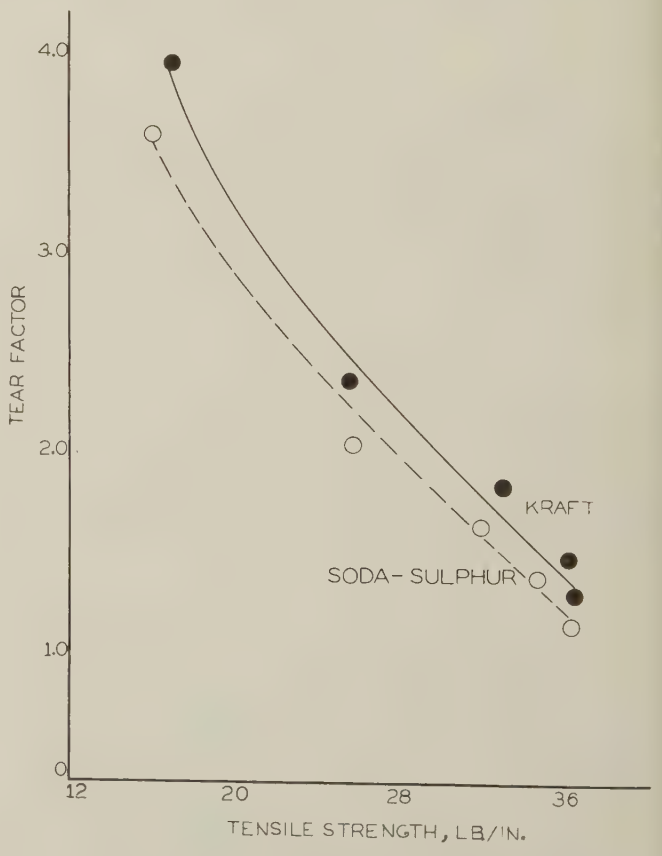

Fig. 1. Tear-tensile relationship of kraft and soda-sulphur pulps

Table II. Physical Properties of Unbleached Pulps

		Cook 1	2	3	4	5	6
Schopper-Riegler freeness, ml.	0[a]	885	890	890	885	890	895
	5	880	880	890	885	890	890
	15	850	865	870	860	870	870
	30	785	780	775	810	800	805
	50	470	475	475	530	535	570
	70	235	220	249	285	305	300
Basis weight (25 × 40—500), lb.	0	44.4	45.4	43.0	43.3	42.7	45.8
	5	44.8	45.9	43.9	45.3	45.8	45.6
	15	44.8	45.4	43.8	45.7	44.8	44.1
	30	44.2	43.4	44.4	45.1	45.3	45.3
	50	47.2	44.8	43.7	43.6	44.8	44.4
	70	44.5	46.9	44.4	46.4	46.8	47.4
Caliper, mils	0	5.2	5.1	5.0	5.4	5.2	5.1
	5	4.8	4.7	4.7	5.0	4.9	4.7
	15	4.1	4.1	4.1	4.3	4.1	4.1
	30	3.8	3.6	3.8	3.8	3.8	3.8
	50	3.6	3.4	3.4	3.4	3.5	3.5
	70	3.2	3.4	3.3	3.4	3.4	3.5
Apparent density	0	8.5	8.9	8.6	8.0	8.2	9.0
	5	9.3	9.8	9.3	9.1	9.3	9.7
	15	10.9	11.1	10.7	10.6	10.9	10.8
	30	11.6	12.1	11.7	11.9	11.9	11.9
	50	13.1	13.2	12.9	12.8	12.8	12.7
	70	13.9	13.8	13.5	13.6	13.8	13.5
Bursting strength, pt./100 lb.	0	30	34	33	26	25	30
	5	58	65	62	59	57	56
	15	103	109	108	104	102	104
	30	127	127	136	134	136	136
	50	147	147	145	157	153	144
	70	150	137	151	150	148	159
Tear factor	0	3.54	3.72	3.49	3.44	2.93	3.54
	5	3.84	3.42	3.62	4.00	3.69	4.25
	15	2.14	2.05	2.05	2.39	2.46	2.36
	30	1.74	1.59	1.71	1.82	1.85	1.92
	50	1.42	1.43	1.37	1.43	1.47	1.60
	70	1.10	1.22	1.22	1.27	1.30	1.43
Tensile strength, lb./in.	0	11.7	11.8	9.9	10.4	9.1	11.5
	5	15.2	17.9	14.8	16.2	16.4	19.9
	15	25.6	27.0	24.2	25.7	23.2	27.3
	30	32.5	32.6	30.1	32.5	32.1	33.6
	50	35.1	34.2	33.7	35.9	35.1	36.8
	70	33.6	36.8	37.5	36.1	34.3	37.9
M.I.T. fold	0	12	16	12	14	11	21
	5	139	172	173	124	121	151
	15	300	325	373	602	543	578
	30	459	513	585	1015	786	824
	50	789	809	950	1332	1166	1164
	70	1065	1083	1008	1472	1255	1672
Zero-span tensile, lb./in.	0	44.3	53.2	39.9	42.5	50.0	48.6
	5	46.5	53.2	47.3	46.8	57.5	49.3
	15	52.5	57.0	46.0	48.5	56.2	46.1
	30	51.5	56.1	49.2	51.4	55.7	49.6
	50	54.0	57.0	47.9	46.7	54.3	49.5
	70	48.6	57.5	47.6	47.0	56.7	48.9

[a] Beating time, min.

level than they are at 400-ml. freeness. The several strength differences indicate that the soda-sulphur cook reduces not only the bonding potential but also the strength of the individual fibers. The strength differences would not, however, be classified as major.

The desirability of pulping with polysulphide-rich liquors is dependent upon several factors. The yield advantage, which was demonstrated experimentally, is offset to some extent by the need for a modified recovery system which would produce a soda liquor and elemental sulphur (or one in which the sulphur is not recovered). In addition, Berthiér noted that corrosion of iron vessels might be expected from this type of cooking liquor.

Waiving consideration of the latter factor momentarily, the polysulphide (or soda-sulphur cook as we will hereafter term it) system can be compared with a conventional kraft system to determine whether the expected yield advantage would justify the necessary modification of the kraft mill.

Assuming that a 2% yield increase (based on wood charged to the digester) can be realized, a soda-sulphur system in a 300-ton mill would result in a production of 312 tons of pulp (300/48 × 50). This represents an advantage of about $1400 per day (based on average price of unbleached kraft pulp). Offsetting this is an increased demand for sulphur since, to produce polysulphides in a liquor equal to the sulphidity afforded conventionally by the monosulphide, an excess of sulphur must be provided to take care of the unavoidable formation of sulphate and thiosulphate. Analysis of the soda-sulphur white liquor, shown in Table I, indicates that for each 100 lb. of sulphur present in the active form, 30.7 lb. of sulphur would be required in the form of sulphate and thiosulphate. (It should be emphasized at this point that the experimental soda-sulphur liquor was made with no intent or effort to minimize thiosulphate formation. Whether or not an optimum set of conditions would afford savings in this direction is not known.) Therefore, a mill processing 624 tons of wood to produce 312 tons of pulp, using the cooking conditions practiced in these experiments, would charge (during a single 24-hr. period) active alkali amounting to 17% of 624 tons, or 106.08 tons (Na_2O basis). Assuming a sulphidity of 30%, 31.82

Table III. Bleaching Conditions and Product Variables

(Polysulphide cooking experiments)

Type of cook →	Cook 1	2	3	Av.	4	5	6	Av.
	Soda-sulphur				Kraft			
Chlorination								
Residual Cl_2 (basis Cl_2 applied), %	13.9	20.8	3.5	12.7	14.3	14.8	16.6	15.2
Caustic Extraction								
Yield after chlorination and extraction, %[a]	94.4	93.5	93.7	93.9	94.1	93.7	92.9	93.6
Hypochlorite Bleachabilities								
Hypochlorite required to reach 80% G.E. brightness, %[a]	2.12	2.16	2.29	2.19	2.30	2.24	2.10	2.21
Total chlorine to 80% G.E. brightness, %[a]	10.7	10.1	11.9	10.9	10.9	10.8	10.4	10.7

Constant Conditions

Chlorinations		Temperature, °C.	40
Chlorine applied, %	10.0	Consistency, %	10.0
Temperature, °C.	20	Time, min.	60
Consistency, %	3.0	Hypochlorite bleachabilities	
Retention time, min.	60	Consistency, %	10.0
Caustic extractions		Temperature, °C.	38
NaOH applied, %	3.0	Time, hr.	4.0

[a] Basis unbleached pulp.

tons of sulphur compounds (as Na_2O) would be present. The sulphur contained in these compounds would be 31.82/62 × 32 or 16.42 tons of sulphur per day. In order to satisfy the sulphur requirements of the soda-sulphur liquor, the sulphur actually used would amount to 16.42 tons × 130.7% or 21.46 tons per day.

connected with the conversion of the H_2S gas to elemental sulphur and the preparation of the soda-sulphur liquor to obtain the maximum polysulphide content are difficult to evaluate at this time. However, if sulphur could be recovered at a cost less than the delivered price of purchased sulphur, the difference

Table IV. Comparison of Physical Properties of Kraft and Soda-Sulphur Pulps

Type of cook →	*Cook* 1	2	3	*Av.*	4	5	6	*Av.*
	Soda-Sulphur				*Kraft*			
	Properties at 700-ml. Schopper-Riegler Freeness							
Beating time, min.	36	37	37	37	37.5	39.5	40.5	39.2
Apparent density	12.3	12.6	12.3	12.4	12.3	12.4	12.4	12.4
Bursting strength, pt./100 lb.	135	142	142	140	146	148	145	146
Tear factor	1.62	1.52	1.40	1.51	1.64	1.63	1.72	1.66
Tensile strength, lb./in.	33.2	33.5	31.7	32.8	34.1	34.3	35.6	34.7
Zero-span tensile, lb./in.	52.0	57.2	48.5	52.6	50.4	56.3	49.0	51.9
Ratio, tensile:zero-span	0.64	0.58	0.65	0.62	0.70	0.61	0.72	0.68
Folding endurance, double folds	583	622	730	645	1155	1022	1030	1069
	Properties at 400-ml. Schopper-Riegler Freeness							
Beating time, min.	55	53.5	54.5	54	58.5	60.5	62	60.5
Apparent density	13.3	13.4	13.1	13.3	13.2	13.3	13.1	13.2
Bursting strength, pt./100 lb.	148	147	149	148	158	154	154	155
Tear factor	1.33	1.40	1.32	1.35	1.34	1.37	1.47	1.39
Tensile strength, lb./in.	35.2	34.8	35.0	35.0	36.2	35.0	37.6	36.6
Zero-span tensile, lb./in.	52.0	57.2	48.5	52.6	47.4	56.1	49.0	50.8
Ratio, tensile:zero-span	0.68	0.61	0.72	0.67	0.76	0.62	0.77	0.72
Folding endurance, double folds	853	853	1000	902	1430	1250	1550	1410

If the recovery system were operated in such a fashion that the sulphur would be entirely lost from the system, then (assuming a price of $32 per ton for sulphur), sulphur make-up would cost about $687 per day. This would leave a net advantage of about $713 per day for the 300-ton mill.

Because of the obvious economic and pollution problems posed by the elimination of sulphur compounds from the recovery smelt, a system which would bring the sulphur back into the operation would be desirable. Since polysulphides are converted to the monosulphide in the digester, the black liquor would be comparable to a kraft black liquor in all respects except for the higher level of thiosulphate content and a lower ratio of organic to inorganic matter. This liquor could be concentrated in the conventional manner and burned in the recovery furnace. The smelt would then be composed primarily of sodium carbonate (Na_2CO_3) and sodium sulphide (Na_2S). Dilution with water would result in a typical kraft green liquor. To remove the sodium sulphide, a stripping tower could be employed using carbon dioxide (probably obtained from the lime kiln) to convert the sodium sulphide to hydrogen sulphide. The H_2S gases given off could be further treated by the Chance-Claus process with air in the presence of a catalyst (Fe_2O_3 or activated charcoal), to produce elemental sulphur and water. The green liquor, freed of the sulphur compounds, would be causticized in the usual manner. The resulting sodium hydroxide solution could be mixed with the elemental sulphur to produce the soda-sulphur cooking liquor.

The costs involved in this recovery are extremely difficult to predict. There are three steps proposed which are not necessary in a conventional kraft recovery, all of which would mean additional expense since none of the usual stages are eliminated. Problems would be added to the previously suggested monetary advantage of $713 per day for a 300-ton mill.

The digester corrosion cited by Berthiér seems to be refuted to some extent by work reported by Hassler (*4*) and Dela Grange (*3*) in 1955. The experience of the West Virginia Pulp and Paper Co. in their Tyrone plant was cited to prove that a soda-sulphur liquor was much less corrosive than one containing monosulphide only. Even mixing sulphur and a solution containing monosulphide acted to make the latter less corrosive. It was stated that polysulphides in very small amounts may be harmful, but if the percentage is quite high, they seem to inhibit the reaction with the metal of the digester. It was recommended that the polysulphides be added at the causticizer.

CONCLUSIONS

The presence of polysulphides in alkaline cooking liquors was found to increase the unscreened yield from southern pine chips by about 1.7%. The screened yield advantage was 2% because of the fewer screenings in the polysulphide pulp.

The polysulphide pulp had the same characteristic color as the kraft pulp and bleaching resistance was the same.

The pulp strength properties of the polysulphide pulps were somewhat inferior to those of the regular kraft pulps.

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Rice Straw for Bleached Papers

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Rice straws from Arkansas, Louisiana, and Egypt were studied as raw materials from which to make fine bleached papers by mechano-chemical and pressure-pulping techniques. Pulping was done with both caustic soda and kraft chemicals. An effective washing treatment was developed to remove friable leaf material and debris from the straws before pulping. No unusual equipment or techniques are required to prepare quality pulps from rice straw. Selected rice straw pulps were converted either alone or in blends with wood pulps to a variety of papers on a laboratory fourdrinier machine.

RICE is grown in many areas of the world which are deficient in timber. As an annually replenished crop residue, rice straw could conceivably satisfy a substantial portion of the increasing demand for papermaking materials. Although about 90% of the approximately 260 million acres of rice lands is concentrated in the Orient and southern Asia, nearly 2.5 million acres are given to rice growing in the United States. Three fourths of this acreage is shared equally by Arkansas, Louisiana, and Texas; one fifth by California, and the remainder by Mississippi (*1*).

Commercial manufacture of paper from rice straw is not a recent development; newspapers printed on rice straw paper were used in England during the early 19th century (*2*). Pulp and paper manufacture at that time, however, was not favored with the economies and technical benefits of modern techniques and equipment. In 1939 commercial pulping of rice straw was attempted on a limited scale near Chico, Calif. (*3*). More recently Lauer and Ghoneim (*4*) reported that soda cooking was more satisfactory than the kraft process for pulping rice straw. In studies on rice straw of British Guiana, Chittenden and Morton (*5*) concluded that the sulfate process was more suitable for pulping.

Conventional pressure-pulping techniques, whether kraft or soda, are not necessarily preferred procedures for processing bulky crop residues. Aronovsky and Lathrop (*6*) found that when pulping was done with 12% kraft chemicals, the yield was 25% greater and the ash content 50% less by the mechano-chemical (M-C) process than by pressure techniques. These benefits, plus the obvious advantage of pulping at atmospheric pressure, warranted investigation of the mechano-chemical process for its applicability in pulping rice straw. Results are presented on the experimental pulping of rice straw by both the pressure and mechano-chemical processes using soda and kraft chemicals, and on the development of a technique for removing hulls, residual grain, leaf materials, and trash from rice straw prior to pulping.

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MATERIALS AND METHODS

Four sound rice straws from three geographic areas were used: One from Arkansas, one from Louisiana, and two from Egypt. Chemical characteristics of these straws were determined according to TAPPI Standards (*7*) except for lignin and MEA cellulose; analytical procedures for these have been described by Aronovsky and Nelson *et al.* (*8*, *9*).

To prepare some of the machine furnishes, typical bleached wood pulps from commercial operations were secured for blending with rice straw pulps. These were: wood sulfite pulp from Washington, aspen groundwood pulp from Wisconsin, and wood soda pulp from Ohio. In addition an experimental bleached reed (*Phragmites communis*) pulp prepared incidental to other studies at the Northern Laboratory was also used for blending. This reed, common to Egypt, had been pressure pulped with 22% kraft chemical.

Straw Preparation

Culm length of all straw was reduced to about 1 to 3 in. by a rotary knife-type cutter. A major portion of the chopped straw was passed over a standard 20-mesh screen to remove fines and dust prior to pulping; this material is subsequently identified as screened straw. The balance of the chopped straw was cleaned in the following manner:

A 3-ft. hydrapulper, equipped with a low-vane rotor and an extractor plate having $^1/_8$-in. diameter perforations, was charged with cold water and sufficient chopped but unscreened straw to make a 10% consistency. The rotor was stopped after 2 min. and the leaf fragments, grain, hulls, dirt, and trash drained away; the culms remained within the hydrapulper. Fresh water was added, and the entire cycle repeated twice. A total of 7 min. was required for the complete treat-

Table I. Proximate Chemical Composition of Four Rice Straws and a Representative Wheat Straw (Values are on Moisture-Free Basis)

Crop source	*Rice*				*Wheat*
	Arkansas, %	*Louisiana*, %	*Egypt*, %		*Illinois* %
Ash	18.8	17.3	17.4	18.7	6.3
Lignin	13.1	12.4	12.0	13.0	16.8
Pentosans[a]	23.4	26.7	28.1	24.9	31.9
Extractives					
Hot water	14.6	15.5	18.1	14.0	10.0
1% NaOH	50.9	52.0	53.7	49.0	41.2
Alcohol-benzene	4.6	5.9	6.0	4.1	2.5
Cross and Bevan cellulose (ash-free)	46.1	43.3	45.9	44.8	54.7
Alpha-cellulose (ash-free)	27.9	25.3	28.1	28.0	37.3

[a] Pentosans = furfural yield × 0.8.

Table II. Physical Composition of Some Baled Straws

	Rice, %	*Wheat, %*	*Barley, %*	*Rye %*
Culms	32.4	68.5	58.8	68.1
Leaf sheaths	33.1	20.3	31.5	19.8
Leaf blades	16.4	5.5	4.9	2.9
Rachises, glumes, and nodes	11.9	4.2	1.8	7.4
Residual grain	0.7	1.0	1.1	1.3
Debris and fines	5.5	0.5	1.9	0.5

ment (3 cycles). Culms cleaned in this fashion are identified as washed straw.

The solid material passing through the extractor plate was collected over a perforated metal screen having $^1/_{16}$-in. diam. openings. These solids also were retained for evaluation in pulping operations.

Pulp Preparation

While investigating the mechano-chemical and pressure processes both caustic soda and kraft chemicals were used in a range of 12 to 18% of the dry weight of the prepared straws. The same amounts of chemicals were not necessarily used in all processes nor with all materials.

Pulping by the mechano-chemical process was carried out in either a 3-ft. hydrapulper (38-in. diam. × 48-in. deep) as described previously (*6*) or in a 5-gal. laboratory unit improvised from a stainless-steel round-bottom mixing bowl and a suspended impeller. Results with the 5-gal. unit are comparable to those of the larger pulper. Pulping periods were 45 and 60 min. Progress in pulping the first charge of rice straw in the 3-ft unit was judged visually and by standard beater tests made on pulp samples removed at 15-min. intervals.

After cooking, pulps were run into a metal drain box having a perforated bottom with $^1/_{16}$-in. diameter openings, washed to remove residual chemicals, and screened over an 8-cut flat screen. Yields of both washed (crude) and screened pulps were determined. Beater and strength characteristics, bleach requirements, and the lignin, pentosan, and ash contents of the screened pulps were determined according to TAPPI Standards (*7*) and other established methods (*8, 9*). A number of screened unbleached pulps were passed through a Centri·Cleaner to remove fine shives and foreign materials. Benefits of centrifugal cleaning were appraised by changes in bleach requirements and in strength characteristics.

Table III. Comparative Physical Characteristics and Bleaching Properties of Mechano-Chemical Pulps from Cleaned Culms and Debris Obtained by Hydrapulper Washing of an Egyptian Rice Straw[a]

Raw material	*Strength characteristics at 500 ml. S.-R.* — *Burst, pt/100 lb.*	*Tensile, G./rm.-lb.*	*Tear factor*	*Folds, Schopper, No.*	*Bleach as required,*[b] *%*
Cleaned culms (washed straw)	109	180		480	6.0
Debris (material passing $^1/_8$-in. diam. perforations retained by $^1/_{16}$-in. diam. perforations)	27	83	21	2	23.5

[a] Both materials cooked 1 hr. with 14% NaOH based on weight of solids.
[b] As chlorine for 70 Hunter brightness.

In pulping by the pressure process all cooking was done in a 10-gal. stainless-steel jacketed digester tumbling at 1 r.p.m. Cooking time was the period at 170°C. and did not include the 20 to 25 min. required to heat the loaded digester up to temperature, nor the 10 min. to drop the pressure to 40 p.s.i. (140°C.) when the loads were discharged into a blowpit. Subsequent treatment of the pulps was the same as for those pulps prepared by the mechano-chemical process.

All pulps used singly or in blends on the laboratory fourdrinier machine (10 in.) were bleached to 70 Hunter brightness in three stages as follows:

(1) Chlorination for 1 hr. with 70% of the chlorine demand at 5% consistency and room temperature.
(2) Extraction with 2% NaOH at 8% consistency for 30 min. at 80°C.
(3) Treatment with calcium hypochlorite equivalent to 30% of the chlorine demand for 3 hr. at 40°C.

Adequate agitation was provided in each stage. A water wash followed each stage with the final wash water containing a small amount of sulfur dioxide.

Machine Papers

All bleached pulps to be incorporated into machine furnishes were passed through a Centri·Cleaner before use. These pulps were also screened on a 4-cut flat screen before running on the laboratory fourdrinier or before blending with other pulps to prepare machine furnishes. Commercial wood pulps, as needed for individual blends, were refined in a laboratory beater to the indicated freeness values, screened over an 8-cut flat screen, and then blended with rice straw pulp or run directly on the paper machine. Bleached pulp from the Egyptian reed *Phragmites communis* was handled in a similar manner. Alum and rosin for sizing were added to the furnishes in the machine chest. Machine papers were evaluated for physical characteristics according to TAPPI Standards (*7*). Experimental papers prepared from rice straw pulps or from blends included bond, writing, newsprint, and glassine grades.

RESULTS AND DISCUSSION

The data in Table I show that the baled rice straws are similar in chemical composition even though they originated in different geographic areas. All samples show the characteristic high-ash content peculiar to rice straws. Lignin, pentosan, and cellulose contents are significantly less than they are in wheat straw that has been presented for comparison; on the other hand, the solubles or extractives are greater. Without further tests it might be concluded that rice straw would produce less pulp. Data on the physical composition of baled cereal straws presented in Table II tend to support such a conclusion; that is, in rice straw leaf materials, nodes, residual grain, and debris are more abundant. The relative amounts of extraneous materials in the baled straw might reflect to some extent the conditions under which they were collected and baled. Removal of these materials at appropriate stages would be desirable. Their deleterious effects in papermaking are indicated by comparing chemical and physical properties of pulps from washed straw and from debris (Table III). Pulp from the debris lacked beneficial physical characteristics, and for comparable brightness

Table IV. Influence of Straw Cleaning Treatments, Pulping Processes and Concentration of Pulping Chemicals on Pulp Yields, Bleach Requirements,[a] Ash Contents and Strength Characteristics in Preparing 70-Brightness Pulps from an Egyptian Rice Straw

Straw-preparation[b]	Chemical concn.,[c] %	Ash in pulp, %	Lignin in pulp, %	Yield: Crude pulp, %	Yield: Screened pulp, %	Bleach as Cl_2 required: Pulp, %	Bleach as Cl_2 required: Straw, %	Yield bleached pulp: Chg., %	Yield bleached pulp: Straw, %	Beating time,[d] min.	Strength characteristics at 500 ml. S.-R. freeness: Burst, pt./100 lb.	Tensile, G./rm.-lb.	Tear factor	Folds, No.
Mechano-Chemical Process: 1 hr. at 98°C.														
Soda Cooks														
S	14	3.9	4.9	53.2	35.3	7.7	6.8	32.3	28.5	26	112	172	36	480
W	14	4.7	5.0	60.6	49.5	6.0	4.7	46.8	36.3	20	110	180	40	480
S	16	4.4	5.4	49.6	34.3	6.6	5.8	31.8	28.1	22	106	172	38	440
W	16	4.8	4.7	60.3	46.8	5.6	4.4	43.5	33.8	20	108	180	37	490
S	18	4.3	3.0	48.0	35.2	5.1	4.5	34.0	33.0	18	107	171	40	450
W	18	5.0	4.1	59.3	44.8	3.9	3.0	43.4	33.7	18	114	182	41	530
Kraft Cooks														
S	14	4.3	5.2	56.6	47.1	11.2	9.9	42.0	37.1	16	92	153	38	630
W	14	6.9	5.3	63.7	53.1	8.4	6.5	48.2	37.4	24	87	155	39	230
Pressure Process: 2 hr. at 170°C.														
Soda Cooks														
S	12	14.6	1.7	56.2	39.4	2.6	2.3	37.8	33.4	42	91	154	50	325
W	12	14.3	2.9	60.0	40.0	1.4	1.1	38.6	29.9	40	96	168	44	320
S	14	14.0	1.8	50.8	37.8	2.3	2.0	36.3	32.0	37	94	154	53	300
W	14	11.4	1.4	51.5	43.4	1.3	1.0	41.7	32.4	39	99	167	50	360
Kraft Cooks														
S	14	15.4	3.6	55.3	46.6	2.3	2.0	44.3	39.1	...	75	136	43	90
W	14	15.8	2.8	56.2	47.7	1.9	1.5	45.3	35.2	45	74	139	38	100

[a] Bleach requirements and yields of bleached pulps are reported on the basis of screened pulp or digester charges and on basis of original uncleaned straw.
[b] Straw preparation: S = dry-chopped and screened over U. S. Standard No. 20 sieve; accepts, 88.3%. W = dry-chopped and washed in hydrapulper (1/8-in. extractor plate); accepts, 77.6%.
[c] Concentration based on dry solids in digester charge.
[d] Time to beat pulp to 500 ml. S.-R. freeness.

(70 Hunter) required almost four times the amount of bleach as pulp from washed straw.

The relative effectiveness of screening and washing as a means for upgrading straw and its derived pulps is shown by the results obtained when lots of an Egyptian rice straw from both cleaning treatments were pulped with soda and kraft chemicals by the mechano-chemical and pressure techniques (Table IV). Data include pulp yields, ash and lignin contents of the pulps, bleach requirements, and strength characteristics of handsheets. For comparable pulping treatments the strength characteristics developed from pulps of screened and washed straws are essentially the same. The mechano-chemical process using caustic soda for pulping was more effective in developing pulp strength than pressure techniques or the use of kraft chemicals. Pressure cooked pulps, however, were more economical in bleach consumption than mechano-chemical pulps. Greater yields were realized from pulping with kraft chemicals than with caustic soda. When bleach requirements and yields of bleached pulps are converted to the basis of original dry solids in bales, it is possible to make comparisons between various cooks for bleach requirements and for yields of bleached pulps. Based on straw solids charged into the pulper or digester, the yields of bleached pulps were greater from washed straw than from screened straw. This means that the effective capacity or output of the pulping equipment is greater for washed straw than for screened straw. Straw culms remaining after the washing treatment are more resistant than leaf material to pulping chemicals and consequently, culms give higher yields of pulps than unfractionated straws. Although straw washing removes some water solubles, attached dirt, and friable plant tissue, this treatment has no significant effect

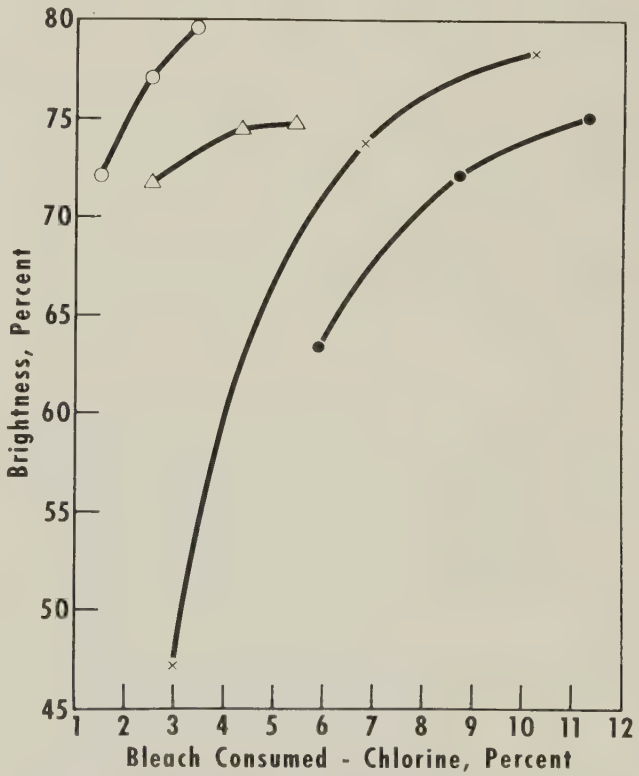

Fig. 1. Hunter brightness values versus bleach consumption for mechano-chemical and soda pulps prepared from an Egyptian rice straw cleaned by screening and by washing. All pulping was done with 14% caustic soda based on dry straw

⊙—Washed straw, pressure-cooked pulp. △—Dry-screened straw, pressure-cooked pulp. X—Washed straw, mechano-chemical pulp. ●—Dry-screened straw, mechano-chemical pulp.

Table V. Influence of Geographic Origin upon the Characteristics of Pulps Prepared from Chopped and Screened Rice Straws by Cooking with 14% NaOH by Mechano-Chemical (M-C) and Pressure (P) Pulping Techniques[a]

Origin → / *Process →*	*Arkansas* MC	*Arkansas* P	*Louisiana* MC	*Louisiana* MC	*Louisiana* P	*Egypt* MC	*Egypt* P
Cooking time, hr.	1	2	3/4	1	2	3/4	2
Ash in pulp, %	5.0	12.1	3.2	3.4	17.1	5.1	18.4
Pentosans in pulp, %	35.0	28.9	32.0	29.1	28.2	30.0	22.4
Lignin in pulp, %	4.7	3.2	5.8	5.9	3.0	7.4	3.8
Crude pulp yield, %	56.7	52.3	52.3	56.1	53.0	52.2	49.1
Screened pulp yield, %	40.6	39.9	35.8	40.1	43.7	43.0	41.8
Chlorine required, %[b]	7.9	5.2	12.3	9.2	2.1	12.1	4.3[c]
Bleached pulp yield, %	36.4	37.0	31.0	32.8	42.9	38.0	38.8
Initial freeness, S.-R., ml.	700	760	705	630	710	630	670
Beating time to 500 ml. S.-R., min.	43	64	21	10	>150	22	32
Characteristics at 500 ml. S.-R.					[d]		
Burst, pt./100 lb.	110	97	115	115	79	95	71
Tensile, g./rm.-lb.	181	165	183	186	140	160	114
Tear factor	36	42	43	46	45	42	40
Folds, Schopper, No.	320	340	400	580	120	270	40

[a] M-C, 3-ft. hydrapulper; P, tumbling digester, 170°C.
[b] Chlorine required for bleaching to 70 brightness, basis dry pulp.
[c] 65 Hunter brightness.
[d] Characteristics at 600 ml. S.-R.; beating time, 92 min.

upon chemical composition of the straw culms nor upon ash content of the pulps in comparison with screened straw. Actually the ash content of the straw is associated chemically with the structure of the culms and leaf material. Retention of the mineral matter throughout the washing treatment is further evidence that it is a constituent part of the rice plant rather than from externally bound dirt and other foreign material. Water solubles and ash content for an Egyptian rice straw after the washing treatment were 8.9 and 15.4%, respectively.

Burst, tensile, and fold characteristics of mechano-chemical soda pulps were essentially the same for concentrations of pulping chemicals at 14, 16, and 18%; M-C soda pulps were superior to those pulps prepared by mechano-chemical kraft, pressure kraft, or pressure soda. Although a maximum yield of bleached pulp was observed for kraft cooking under pressure, the strength characteristics of the resulting pulp were minimum for the group. Based on overall data, caustic soda is the preferred chemical to use with rice straw in the mechano-chemical operation.

The bleach requirements, calculated as per cent chlorine, to develop indicated Hunter brightness values in pulps prepared by cooking both screened and washed Egyptian straws with 14% caustic soda by both M-C and pressure techniques are shown in Fig. 1. Substantial improvement in brightness is achieved for a given amount of chlorine when washed rather than screened straw is used.

Relative ease of pulping and suitability of these rice straws for papermaking was appraised by pulping all straws with 14% caustic soda by both techniques. Data showing processing conditions and the physical and chemical characteristics of soda pulps are presented in Table V. Differences in pulp yields, initial freeness, and beating times to 500 ml. S.-R. freeness indicate variations expected in pulping and refining straws of different geographic origin. To what extent variety might influence these results is not known. Pulps prepared by M-C techniques in all cases had higher lignin and pentosan contents and lower ash content than pulps from pressure-cooked straws. Strength characteristics (tear excepted) of the mechano-chemical pulps were greater than those from the pulps prepared by the pressure treatment. In this series of tests the Egyptian straw pulps developed the lowest physical characteristics. However, data in Table IV show that under selected conditions Egyptian rice straw can be pulped and refined to have characteristics comparable to those of Arkansas and Louisiana rice straw pulps.

Effectiveness of centrifugal cleaning on physical characteristics and bleachability was appraised with pulps prepared from Louisiana and Egyptian rice straws, the former washed and cooked with 16% caustic soda and the latter dry-screened and cooked with 14% kraft chemical (Table VI). Although the original Egyptian pulp appeared inferior strengthwise to the Louisiana pulp, both improved to about the same level as a result of the cleaning treatment. A number of machine furnishes, prepared from rice straw pulps or blends, were run on a 10-in. fourdrinier machine. Composition and physical evaluation data for the experimental papers are shown in Table VII. While the data are insufficient to make valid comparisons between the different furnishes, they illustrate in a general way the characteristics of several machine papers prepared from straw pulp alone and in blends with other pulps. In some instances the

Table VI. Physical and Chemical Characteristics of Rice Straw Pulps Before and After Centri-Cleaning

	Strength characteristics at 500 ml. S.-R. freeness				*Bleach as Cl_2 required, %[a]*
	Burst, pt./100 lb.	*Tensile, G./rm.-lb.*	*Tear factor*	*Folds Schopper, No.*	
Louisiana, washed, 16% NaOH					
Original	101	174	44	430	6.1
With Centri-Cleaning	113	190	46	550	5.6
Improvement, %	12	9	5	28	8
Egyptian, screened, 14% kraft					
Original	92	153	38	490	11.2
With Centri-Cleaning	115	190	43	630	...
Improvement, %	25	24	13	29	...

[a] For 70 Hunter brightness.

Table VII. Composition and Characteristics of Experimental Bleached Papers Prepared on a 10-in. Fourdrinier Machine from Rice Straw Pulps, Singly and in Blends, with a Commercial Wood Sulpfite Pulp and with an Experimental Reed Pulp

Machine furnish			Freeness, S.-R.	Basis weight	Physical characteristics								
Straw parts	Wood parts	Clay[a] parts	straw/wood, ml.	25 × 40—500, lb.	Opacity, %	Density, g./cc.	Brightness, %	Burst pt./100 lb.	Tensile,[b] G./ream-lb. MD	Tensile,[b] G./ream-lb. CD	Tear factor MD	Tear factor CD	Fold, Schopper, MD	Fold, Schopper, No. CD
Screened Louisiana Rice Straw—14% Soda M-C Pulp														
100[g]	0	0	190	32	61	0.84	70	102	203	144	36	42	1285	861
100	0	15	520/...	47	88	0.69	72	76	133	104	57	58	196	95
75	25[c]	10	585/650	49	91	0.58	75	58	143	78	85	95	185	32
50	50[c]	10	625/625	44	90	0.59	72	69	179	83	80	96	407	49
0	100[c]	10	.../700	45	88	0.62	69	50	152	54	92	132	387	20
Washed Louisiana Rice Straw—16% Soda M-C Pulp														
100[h]	0	0	240	35	64	0.80	72	90	205	130	29	29	708	304
100[d]	0	10	600/...	44	89	0.52	78	58	131	77	69	81	61	16
100[e]	0	10	600/...	38	88	0.52	77	44	138	76	71	96	41	10
75	25[c]	10	700/600	47	92	0.56	75	59	134	72	79	95	87	24
50	50[c]	10	600/650	55	91	0.62	78	58	144	87	99	106	321	82
0	100[c]	10	.../700	45	88	0.62	69	50	152	54	92	132	387	20
Screened Egyptian Rice Straw—14% Soda M-C Pulp														
100	0	15	600/...	44	91	0.60	74	58	140	83	60	72	75	19
50	50[f]	0	500/650	45	86	0.56	72	65	164	91	65	72	118	38
0	100[f]	10	.../600	44	83	0.50	76	37	99	57	65	78	142	5

[a] Plus rosin and alum.
[b] MD = machine direction; CD = cross direction.
[c] Commercial bleached softwood sulphite pulp.
[d] Pulp screened through 6-cut flat screen.
[e] Pulp screened through 4-cut flat screen.
[f] Experimental reed pulp (*Phragmites communis*), −22% kraft.
[g] Greaseproof.
[h] Manifold.

strength properties of the blends are superior to those of either component alone. Two characteristics that were observed, but to which numerical ratings could not be assigned, were sheet formation and surface smoothness. In the straw blends these characteristics were better than in the all-wood sheets, and these, as well as improvements in strength characteristics, were attributed to the high value of the length-to-diameter ratio (170:1) of the rice straw fibers although only about 1450mμ in length. These observations with rice straw pulps are in agreement with those of earlier workers who studied blends of wheat straw pulp and wood pulp for a variety of papers including magazine, book, bond, and greaseproof types (*10*).

SUMMARY AND CONCLUSIONS

Rice straws from Arkansas, Louisiana, and Egypt were investigated as raw materials for the preparation of fine bleached papers. Pulping was done by both mechano-chemical and pressure techniques using caustic soda and kraft chemicals as pulping agents. Experimental pulps were run singly and in blends with commercial wood pulps on a laboratory fourdrinier machine.

An effective washing treatment was developed to remove leaf material, hulls, grain, and trash from straw culms. In contrast to dry screening, washing increases the efficiency of pulping and bleaching operations, but does not significantly alter pulp yield based on original uncleaned straw.

Within the scope of the study the following conclusions are made:

1. Rice straws from Arkansas, Louisiana, and Egypt can be used to make practical bleached pulps of essentially the same quality for papermaking. Processing conditions need to be modified to the specific straw.

2. To produce bleached pulps of good physical characteristics, the mechano-chemical technique with caustic soda was more effective than the pressure technique; however, lower lignin content and better bleaching properties were realized with pressure pulping.

3. Rice straw pulps of low ash content (<5.0%) can be prepared by mechano-chemical techniques with caustic soda, whereas high values (>12.0%) are obtained by pressure treatments or with kraft chemicals.

4. Centrifugal cleaning is an effective means for improving quality of rice straw pulps with respect to both bleachability and strength characteristics.

5. Supplementation of rice straw pulps with long-fibered pulps is desirable when fine surface characteristics and resistance to tear are important.

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Chemical Modification of Papermaking Pulps

KYLE WARD, JR.

Cellulosic fibers can be partially substituted with hydrophilic groups so as to produce fibrous derivatives of low degree of substitution (D.S.) which produce paper sheets of improved strength properties. This paper gives a brief summary of developments in this field within the past few years. Many different types of groups have been introduced into the cellulose molecule, but the two investigated most thoroughly are the carboxymethyl and the hydroxethyl group. In order to be suitable for paper the fibers must have the property of insolubility in water. Therefore, the D.S. is kept quite low; even at such low D.S. as 0.03 or 0.04, however, marked increases in tensile strength, bursting strength, and folding endurance are observed. This does not occur at the expense of tearing strength, as is frequently the case when the other strength characteristics are improved by mechanical refining, for the tearing strength also increases somewhat. Opacity, however, is decreased. A hypothesis is proposed to account for the behavior of this type of fiber. In short, it is suggested that the chemically modified fiber has, as it were, a beater additive built into its structure. This results in more rapid response to mechanical refining and the development of greater bonding strength. A comparison of some effects of beater additives and of chemical modification is given to confirm this hypothesis.

It is of interest that cellulose derivatives were prepared before cellulose itself was isolated and given a name. At least Braconnot's preparation of nitrated cotton in 1833 (*1*) was several years earlier than Payen's isolation of the cell wall material which he called cellulose (*2*). The preparation of cellulose derivatives became the basis for several industries.

Not until relatively recently, however, was it recognized that high substitutions were not necessary in order to effect large differences in the properties. For a long time the only derivatives investigated were those in which the relatively insoluble native cellulose was brought into solution so that it could be handled in the form of a liquid. It has recently been recognized that the substitution of only a few of the hydroxyl groups in the cellulose molecule may produce extremely important changes in the physical properties of fibers without loss of the frequently valuable property of insolubility and without disturbing the morphological structure of the fiber.

It is not, strictly speaking, true to say that this development is recent. Reviews of the use of chemical modification of cellulose for textile purposes have recently been made by Buras (*3*) and by Fisher and Perkerson (*4*). From both of these reviews, it is clear that the idea of insoluble fibrous modifications goes back half a century although the commercial development of special cases is recent. As Buras points out, the first process was probably partial acetylation by Cross (*5*). However, Cross did not apply this to commercial use until 1907 (*6*), if we take patents as an evidence of attempting to apply a process commercially. In this case the earlier patent of L. Lederer in 1902 takes priority (*7*). Vignon obtained a still earlier patent for introducing nitrogen into the cellulose molecule in order to change the dyeing characteristics (*8*), but later workers have shown that this process of treating cellulose with ammonia or ammoniacal solutions does not introduce nitrogen (*9*). However, the application of chemical modification to papermaking fibers came much later, and it is this field which I should like to discuss today.

Jayme and Froundjian in 1940 showed that methylated pulps have improved affinity for water and also improved strength characteristics (*10*). Later, Bletzinger (*11*) studied the acetylation reaction and found that acetylation above 9% increased the water resistance so much that poor bonding was obtained and satisfactory sheets could not be formed from water. However, acetyl values below 6% allowed greater ease of hydration and improved strength was observed in the handsheets. These results were corroborated by Aiken (*12*) and Harrison (*13*).

Although methylcellulose and cellulose acetate at these substitutions have more affinity for water then cellulose, this is due to a decrease in the lateral order and not to solvation of the added substituent group which is less hydrophilic than the hydroxyl. The application of chemical modification became more promising when hydrophilic groups were introduced into the molecule.

Richter in 1936 (*14*) patented the formation of paper from alkali-soluble hydroxyethylcellulose and A. W. Schorger in 1950 (*15*) the preparation of stereotype mats from hydroxyethylcellulose of a degree of substitution of 0.25 to 1.5, which was therefore also in the alkali-soluble range. Walecka in 1956 was the first to show that pulps chemically modified to extremely low degrees of substitution with hydrophilic reagents and still insoluble in any solvents except those for cellulose were superior for papermaking (*16*). It is the developments in this particular field since that time which the author wishes to discuss.

Walecka's work was done at The Institute of Paper Chemistry in partial fulfillment of the requirements for his Doctor's thesis. A great deal more work has been done since at the Institute, both in student research and in cooperative research for various paper companies. At the same time and independently, several pulp suppliers have been interested in this problem and various chemically modified cotton linters pulps are now on the market. The development of one such pulp was recently described by Harpham (*17*). What the author should like to present is a unified picture of published work in this field and of certain unpublished aspects of the work at the Institute.

Walecka and, later, his fellow student, Talwar (*18*), carboxymethylated rag pulps. Such treated pulps produce stronger sheets than the untreated rag pulps and also refine much faster (Fig. 1). Up to about 0.06 degree of substitution, there is a rapid increase in

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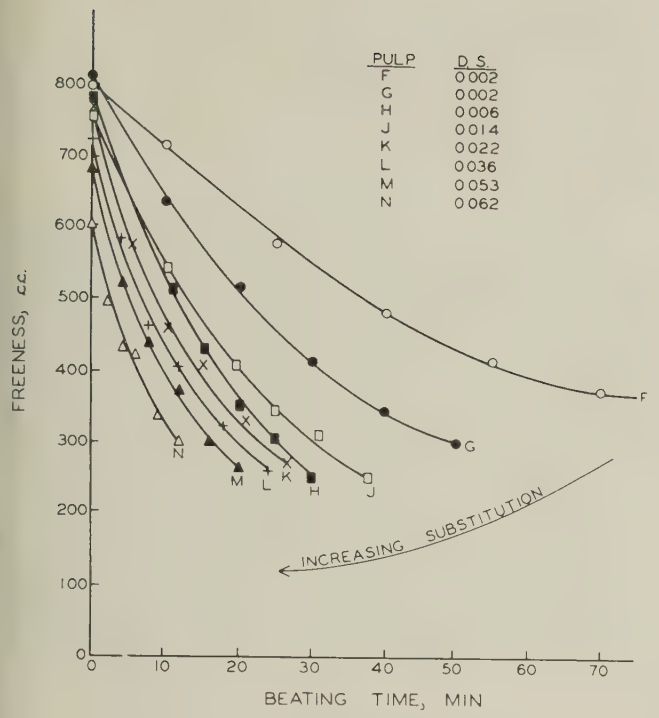

Fig. 1. Freeness versus beating time for carboxymethylated pulps

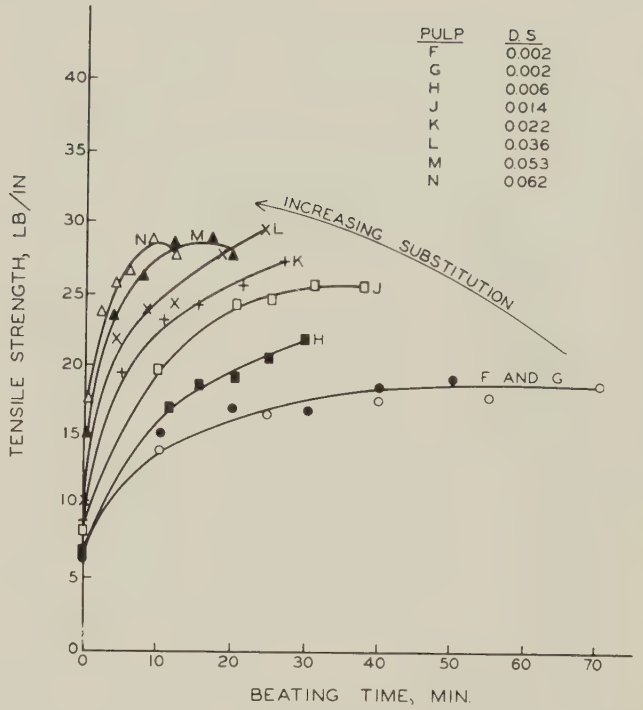

Fig. 3. Tensile strength versus beating time for carboxymethylated pulps

the burst (Fig. 2) and tensile strengths (Fig. 3) and a slight increase in the tearing strength (Fig. 4). Walecka observed an increase in the zero-span tensile strength of the fibers (Fig. 5). Normally, this indicates an increase in the fiber strength. However, Talwar, measuring individual fiber strength directly, was unable to detect any change due to carboxymethylation. The opacity of handsheets decreases as the degree of substitution of the pulp increases (Fig. 6) and so does the porosity. As would be expected, the equilibrium moisture content of the treated pulps is higher (Fig. 7). Photomicrographs show that the chemically treated material consists of more transparent fibers than does the untreated pulp (Figs. 8 and 9). This, of course, is reflected in the observed changes in opacity just mentioned.

You may be interested at this point in knowing

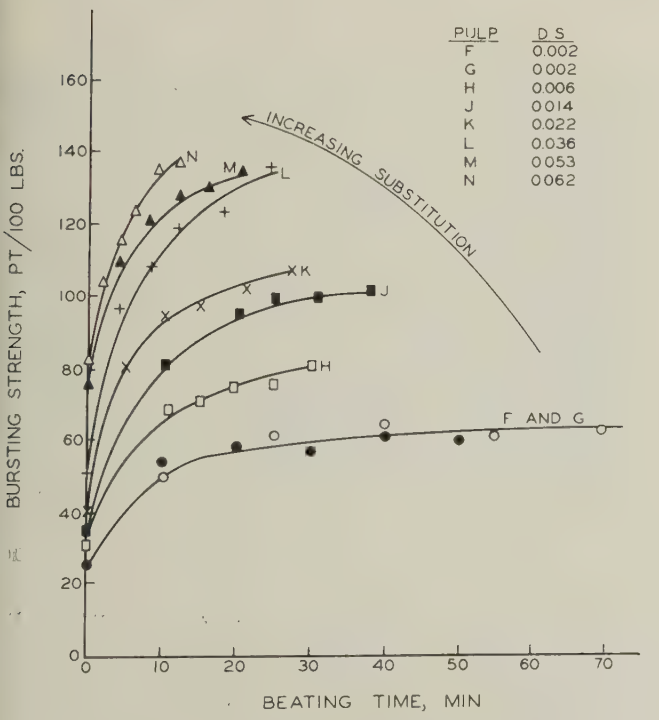

Fig. 2. Bursting strength versus beating time for carboxymethylated pulps

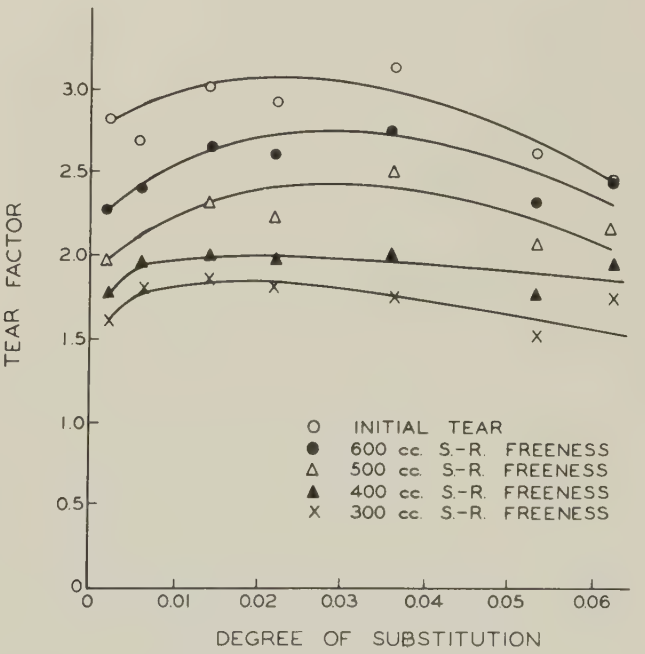

Fig. 4. Tear factor versus D.S. for carboxymethylated pulps

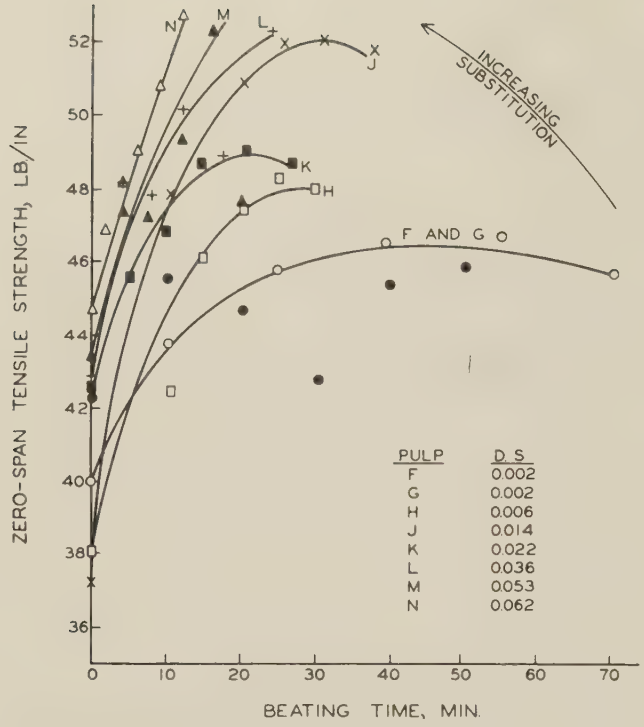

Fig. 5. Zero-span tensile versus beating time for carboxymethylated pulps

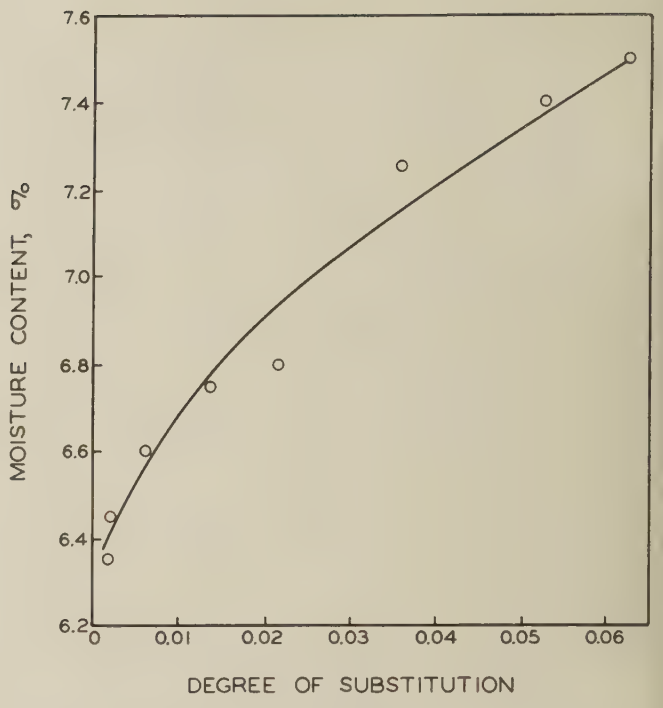

Fig. 7. Moisture content versus D.S. for carboxymethylated pulps

whether or not we are safe in saying that this effect is general and can be obtained with more than one type of hydrophilic group. There seems little doubt of this. Harpham (*17*) has described cyanoethylation, carboxymethylation, hydroxypropylation, and hydroxyethylation to produce improved papermaking properties. Spadaro, *et al.* (*19*), of the Southern Regional Research Laboratory, have also described hydroxyethylated and cyanoethylated fibers. At The Institute of Paper Chemistry other treatments have been studied and promising results obtained on treatment with acrylamide and with β-propiolactone, for example.

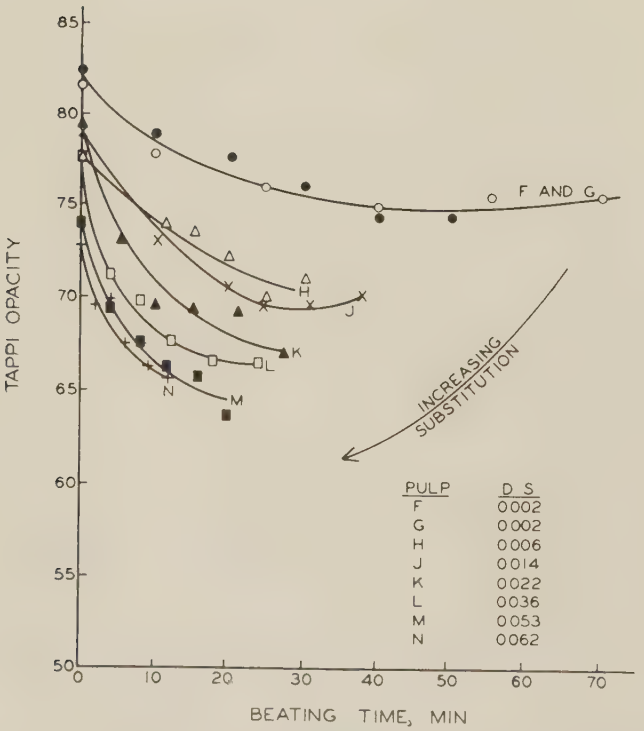

Fig. 6. Opacity versus beating time for carboxymethylated pulps

It is perhaps of interest also to mention treatments which have not given improved papermaking properties. The Crane Co. and the Southern Regional Research Laboratory (*19*) have investigated decrystallization with ethylamine, and have found that there were actually decreases in sheet strength. This is not, strictly speaking, a chemical modification, of course.

At the Institute of Paper Chemistry many chemical treatments proved unsuitable, notably those which produced degradation of the cellulose, such as certain oxidations, and those which might presumably have produced cross linking, such as reactions with aldehydes. Neither of these negative results is particularly surprising.

Walecka, as we have just seen, observed continual increase in the strength properties with increasing degree of carboxymethylation. We have carried out similar studies on hydroxyethylation and obtained similar results (Fig. 10). The data of Spadaro (*19*) are not comparable, having been obtained after Valley refining rather than Jokro, as ours were, and also covering, in general, higher degrees of substitution.

Similar results are obtained with tensile strength (Fig. 11). Tear has been shown both by Harpham (*17*) and Spadaro (*19*) to increase with increasing D.S. at the early stages of beating, but to tend to decrease at low freeness.

We now come to the following important consideration. Why does chemical modification produce increases in the strength of the sheets from such fiber? The mechanism proposed by Talwar for carboxymethylcellulose seems valid and would also satisfy the observations on hydroxyethylcellulose by the various in-

vestigators. This is briefly as follows. When hydrophilic groups are introduced, the fibers have much greater affinity for water, particularly in the modified regions. This is partly due to the fact that any substituent opens up the structure, but also to the affinity of the substituted hydrophilic groups themselves. In

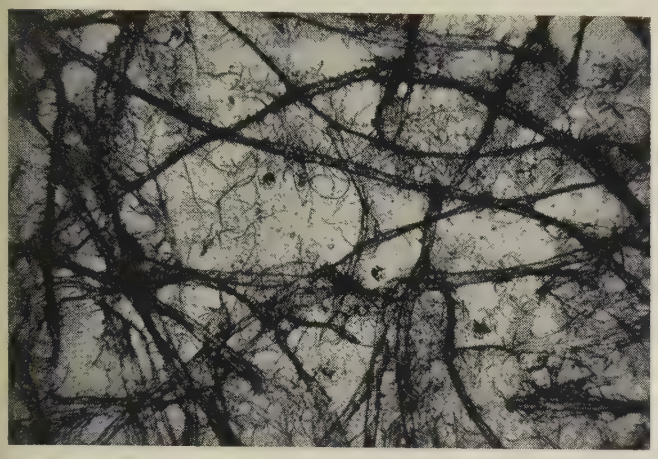

Fig. 8. Untreated rag pulps 160× beaten to 320 S.-R. freeness

this way the wet fiber will be studded with small regions where water has penetrated and separated the molecules by solvation and swelling. At these points there is increased mobility of the molecular segments and increased flexibility of the wet fiber. This persists during the early stages of drying, improving the capacity to form hydrogen bonds both within the fiber and between fibers, for the hydroxyl groups draw closer together on drying because of movement of the molecules and deformation of the fibers. In addition to this effect, the moisture affinity of the fiber is such that the moisture content under testing conditions is higher than for the untreated fiber due to increased moisture regain. Testing at this higher moisture content will produce

Fig. 9. Carboxymethylated rag pulps 160× D.S. 0.062 beaten to 300 S.-R. freeness

somewhat higher folding endurance for this reason alone.

There is a great deal of evidence to support such a theory. Both Walecka and Talwar have presented evidence to show that there will be localized areas of relatively high substitution in a given sample. Various

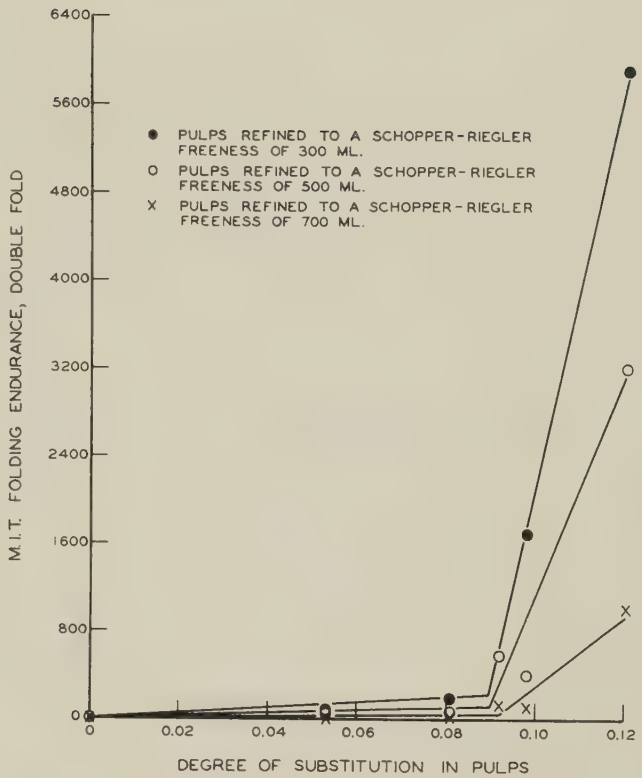

Fig. 10. Folding endurance versus D.S. for hydroxyethylated linters

items that bear on this are the slight lowering of D.S. during the process of sheetmaking, attributed by Walecka to solution of high D.S. material; the observa-

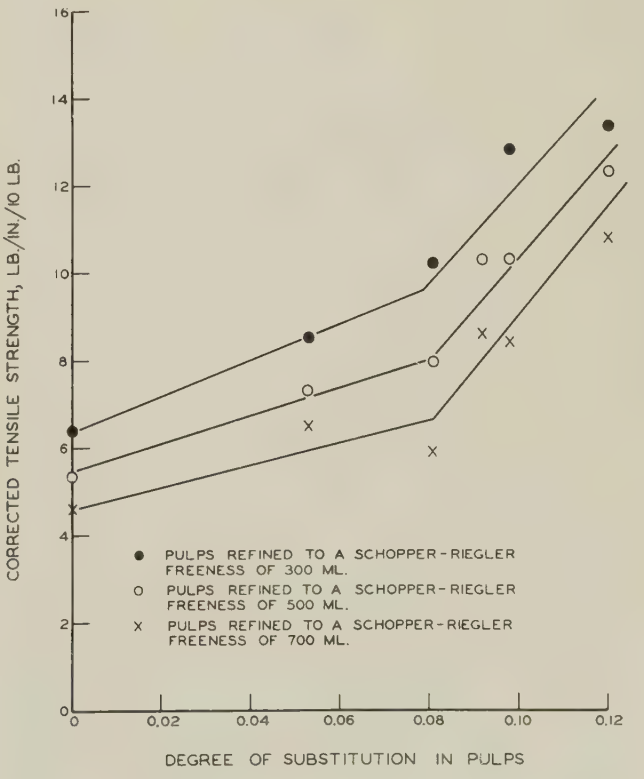

Fig. 11. Tensile strength versus D.S. for hydroxyethylated linters

tion of Talwar that at a pH of 8.5 a certain amount of the material is actually peptized and that this has a higher degree of substitution than the rest; and unpublished work by Morak at the Institute on hydroxyethylcellulose showing that a certain amount of highly substituted material present in these samples is removable by extraction with alcohol, lowering the moles substituted (M.S.) from 0.29 to 0.27. Into such regions water could penetrate very readily to produce the effects

ethylated material suffers in fiber strength, partly by degradation during reaction which, while slight, would be unavoidable and partly, in all probability, by loss of dissolved material. However, the hydroxyethylated material furnishes most of the bonding strength for the sheet. It furnishes such bonding strength, by mechanisms discussed earlier, in mixtures and in uniformly modified fibers, but in the mixtures another factor appears. In our experiments, the blend is beaten as a

Table I. Comparison of Hydroxyethylated and Untreated Cotton Linters

	Untreated			*Treated*			
Refining time, min.	79	127	162	30	90	120	151
Schopper-Riegler freeness, ml.	730	320	225	845	590	365	255
Bursting strength, pt./100 lb.	98	160	156	117	194	213	245
Tear factor	4.56	3.63	3.49	6.49	4.85	4.37	4.01
M.I.T. folding endurance, double folds	18	65	166	34	382	645	1044
Thwing-Albert formation, units	39.2	43.6	35.6	22.5	30.0	31.8	31.2
Corrected tensile strength, lb./in./10 lb.	6.2	8.6	9.6	5.6	9.3	10.0	11.1
Corrected minimum wet tensile strength, lb./in./10 lb.	0.32	0.53	0.32	0.37	0.37	0.37	0.45
Corrected zero-span tensile strength, lb./in./10 lb.	19.1	20.1	20.3	21.2	21.1	21.5	21.1
VVP bonding strength, kp.cm./sec.	3.46	6.08	7.09	4.75	41.0	50.3	51.0

which we are postulating. This improved hydration should, therefore, result in more rapid development of freeness with beating and in more bonding.

Part of the bonding may be within the fiber; Walecka found that carboxymethylated fibers are more transparent fibers. Recent work at the Institute shows that both the fibers and the sheets are more transparent in the case of hydroxyethylcellulose.

One measure of bonding strength is the so-called "viscosity-velocity product." In the I.P.C. measurement of bonding strength, steel rollers carrying films of viscous oil are rolled over test specimens with uniform, but continuously variable, speed. The critical speed, that above which the test specimens suffer open rupture, is determined for each of various liquids of different viscosity. The product of velocity times viscosity has been shown to be constant and this product is taken as a measure of bonding strength. Our measurements have shown that this so-called VVP value of hydroxyethylated fibers increases rapidly with beating and correlates with the higher strength values, particularly the high fold values. Formation is very little changed and fiber strength (zero-span tensile) just as little by hydroxyethylation (Table I).

The usual method of measuring relative bonded area proved unsuitable for use with chemically modified fibers. It is possible that this determination, which depends upon the number of bonds, is increased in the same proportion as the aggregate bonded strength, but we have not shown this directly by experiment as yet.

Interesting data compatible with the theories of Walecka and Talwar have also been derived in a study of mixtures of hydroxyethylated cellulose fibers with untreated fibers. Mixtures of various average M.S. were prepared by blending untreated linters with samples of M.S. about 0.1. If these mixtures were compared with treated linters of the same average M.S., strength properties of the mixtures were considerably higher than those of the uniformly hydroxyethylated fibers. This, it seems to us, is in agreement with the previous hypothesis for the following reason.

The fiber strength of the sheet is being furnished in greater part by the unreacted material, for the hydroxy-

whole, so that the hydroxyethylated fibers, which beat more rapidly, have reached a lower level of freeness in the mixture than has the unmixed fiber. Since our comparisons are made at comparable freenesses, we actually have our bonding material operating at a more advanced stage of mechanical refining in the mixture than in the homogeneous material.

The wet tensile strengths of the treated cellulose are also in accord with the preceding theories. From Table I, it will be seen that although hydroxyethylation improves dry tensile strength, it does not affect wet tensile strength. This would be expected from the affinity for water and the ability of water to disrupt the bonding.

Notice that this picture of strong fibers bonded together by inherently weaker fibers (or inherently weaker material, for it need not actually be fibrous at all) is exactly that of a beater additive. We should like to have a more direct means of checking this and are working on the problem at present, but the data which we have now are in accord with the hypothesis that a chemically modified fiber in papermaking practice behaves as if we had produced a built-in beater additive with no problems regarding retention or subsequent loss of properties.

The essential difference between the sheet properties of the chemically modified pulps and of beater-additive treated pulps is the tear factor. As Harpham (*17*) has pointed out, the increased tear factor resulting from chemical modification suggests a fiber substitution value rather than power substitution. This may arise from increased extensibility of the treated fiber which might, for the same degree of bonding, result in an increase in the ultimate fiber strain and hence in an increase in the energy required to rupture fibers (*20*). Further investigation is necessary to validate or invalidate this hypothesis.

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The Leucoanthocyanin from Black Spruce Inner Bark

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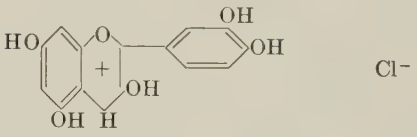
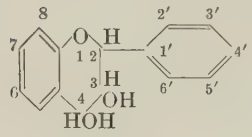
The leucoanthocyanin from black spruce inner bark was isolated through countercurrent distribution followed by precipitation with neutral lead acetate. The anthocyanidin produced by acid treatment of the isolated compound was identified as cyanidin. The leucoanthocyanin 3′,4′,5,7-tetrahydroxy-flavan-3,4-diol was synthesized by the alkaline sodium borohydride reduction of dihydroquercetin and was compared with the isolated compound. Both compounds showed identical infrared spectra and nearly identical ultraviolet spectra. Acetylation of the crude extract from which the leucoanthocyanin was isolated yielded a crystalline product which was identified as 3,4′-dihydroxy-3′-methoxy-stilbene-5-O-β glucoside hexaacetate. The synthetic leucoanthocyanin was used to standardize the propanol hydrolysis test and to extend the concentration range in which consistent results were obtained. Using this standard the leucoanthocyanin was estimated to constitute 10% of the original extract.

The presence of leucoanthocyanins in plants has been known, but only recently have the structures of some of these compounds been determined. Formerly, these compounds were defined by Rosenheim (*1*) as materials producing anthocyanidinlike colors when treated with hot dilute acid. The first thorough investigation of the leucoanthocyanins was carried out by the Robinsons (*2–5*), who determined that these materials were quite widespread and that the majority of them yielded cyanidin (I). Bate-Smith (*6–7*) extended this investigation using paper chromatography, and found that the leucoanthocyanins were widely distributed, particularly in woody plants, and in agreement with the Robinsons found that cyanidin was the most commonly produced anthocyanidin.

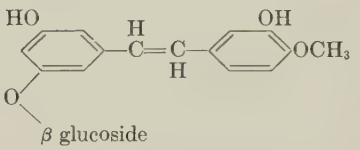
The basic structure of the leucoanthocyanins remained undetermined until King and Bottomley (*8*) isolated and identified a substituted flavan-3,4-diol (II) with the properties of a leucoanthocyanin. They proposed that the flavan-3,4-diol represented one of the basic structural types for the leucoanthocyanins. Keppler (*9, 10*) supported this view by isolating and characterizing a leucoanthocyanin with the same basic structure, but with a different hydroxylation pattern. This basic structure however, does not explain the properties of all the isolated leucoanthocyanins. Several of these compounds have been isolated which were polymeric in nature (*11–13*) and although they yielded anthocyanidins in the usual manner, they were not clearly related to the flavan-3,4-diols. Many leucoanthocyanins have been isolated which yielded the same anthocyanidin (*11–13*), but which did not appear to have the same basic structure. These compounds may be polymers of different molecular weight or they may be stereoisomers which are separable chromatographically.



(I)
Cyanidin chloride



(II)
Flavan-3,4-diol



(III)
Andrews' stilbene glucoside

The yield of anthocyanidin from an acid-treated leucoanthocyanin was always low (10 to 20%). Originally this fact was believed to be due to the difficulty in its isolation (*5*). More recently, chromatographic investigations of the products of an acid-treated leucoanthocyanin showed that the anthocyanidin was actually a minor reaction product (*14, 15*). When the reaction was run in an aqueous medium, the major product was a reddish-brown phlobaphenelike precipitate. This precipitate has been postulated as the product of the acid-catalyzed condensation of the

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flavan-3,4-diols (*12*). Freudenberg and Weinges (*16*) have found that this condensation is characteristic of hydroxyflavans and that condensation is enhanced by hydroxylation in the 4′ and 7 positions whereas hydroxylation of the heterocyclic ring has little effect on the condensation.

The mechanism of conversion of the leucoanthocyanins into anthocyanidins is uncertain. In some cases oxygen is not required for conversion (*2*), while in others it appears to be essential (*12*). In the former case a possible mechanism is a disproportionation leading to the simultaneous formation of a flavan-3-ol (catechin) and the anthocyanidin. The presence of catechin in the reaction products was noted by Forsyth (*11*). On the other hand, catechin has not been detected in the reaction products from an acid treatment of known flavan-3,4-diols (*12*). These facts indicate that all leuconathocyanins may not be simple flavan-3, 4-diols, and that the mechanism of conversion may not be the same for all of these compounds. This means that the term leucoanthocyanin is not defined sharply.

Prior to the demonstration of the flavan-3,4-diol structure for certain leucoanthocyanins, Pigman and co-workers (*17*, *18*) detected the presence of a leucoanthocyanin in black spruce inner bark. Further investigation of the inner bark led to the isolation of a material believed to be a fairly pure leucoanthocyanin. Attempted characterizations through hydrolysis and analysis of methylated and acetylated derivatives indicated the presence of a glucoside which contained several phenolic and alcoholic hydroxyls plus one methoxyl group. However, the complete structure of this compound was not determined.

Recent investigations of various spruce barks have lead to the isolation of two stilbene glucosides (*19–23*). Andrews (*19*) isolated a substituted stilbene glucoside (III) from white spruce bark with properties similar to the material obtained by Pigman, *et al.* (*18*), and which yielded an acetate similar to the acetate obtained by Pigman. Andrews (*19*) and Hoffman (*20*) proved the structure of this compound by degradation and synthesis.

In view of the recent advances in the chemistry of the leucoanthocyanins, which indicated the complexity of this class of compounds, a more complete understanding of the black spruce inner bark leucoanthocyanin appeared desirable. Since only limited evidence concerning the nature of this compound was available, this research was undertaken to characterize this leucoanthocyanin more completely.

PREPARATION AND FRACTIONATION OF THE EXTRACT

The black spruce (*Picea mariana* BSP) inner bark samples were collected from trees felled in the Ripco Experimental Forest near Rhinelander, Wis., in June, 1957. Three trees of uniform size and averaging 38 years of age were felled, the branches removed, and the stems cut into 2-ft. bolts. The outer bark from each bolt was removed and discarded. The soft sticky inner bark was peeled from the bolts, cut into short strips, and placed in jars containing methanol, which served to arrest enzyme action and as the extracting agent.

Table I. Yields of the Fractions from the Methanol Extract

Fraction	*Yield based on inner bark, %*[a]	*Yield based on total extractives, %*
Water-insoluble materials	1.2	7.3
Salt-precipitated materials	6.9	42.0
Ethyl acetate-extracted materials	1.6	9.6
Materials remaining in the saturated salt solution	6.8	41.1
Total	16.5	100.0

[a] Yields calculated on an ovendry unextracted basis.

The extraction was allowed to continue for 2 weeks at room temperature, since hot extraction had been shown to change the nature of the extract (*18*). The methanol solution containing any water removed from the bark was concentrated *in vacuo* until all the methanol was removed. A brownish-colored precipitate separated and was removed by filtration, dried, and weighed. The aqueous solution was diluted with water and saturated with sodium chloride, whereupon a reddish-brown gummy precipitate formed. The entire mixture was filtered until a clear filtrate was obtained. The residue on the filter pad was extracted with methanol and the extract evaporated to dryness and weighed. The clear filtrate was extracted exhaustively with ethyl acetate. The resulting ethyl acetate solution was dried over sodium sulphate and sodium bicarbonate, concentrated to a small volume, dried again with sodium sulphate, and taken to dryness under reduced pressure.

This fraction, subsequently termed the "ethyl acetate fraction," was used as the starting material for the isolation of the leucoanthocyanin. Previously, Pigman, *et al.* (*18*), had prepared and fractionated an extract in a similar manner and referred to the "ethyl acetate fraction" as the "black spruce inner bark leucoanthocyanin." The yields of the various fractions of the methanol extract are given in Table I.

In order to resolve the complex mixtures encountered in this work, two-dimensional paper chromatography was used. The chromatograms were developed first in 6% aqueous acetic acid and then in secondary butanol saturated with water. The chromatograms were examined under ultraviolet light and then sprayed with diazotized *p*-nitroaniline (*24*) or with a mixture of *n*-propanol and hydrochloric acid. The latter spray, developed for the detection of leucoanthocyanins, was prepared by adding 0.5 ml. conc. HCl to 20 ml. of *n*-propanol. The chromatogram was sprayed with this solution and placed in an oven at 105°C. for about 2 min. The leucoanthocyanin developed a faint but distinct pink color and *d*-catechin developed a bright yellow color.

Two-dimensional paper chromatograms of the ethyl acetate fraction revealed spots for at least 12 compounds. One of these spots was identified as *d*-catechin by comparison with chromatograms of an authentic sample of *d*-catechin. The leucoanthocyanin appeared to be a relatively minor component of this fraction.

CHARACTERIZATION OF THE ETHYL ACETATE FRACTION

Aqueous Acid Hydrolysis

Pigman, *et al.* (*18*), reported that the ethyl acetate

fraction on hydrolysis yielded 40% reducing sugar, and demonstrated that the sugar was glucose by forming the phenylosazone and comparing it with authentic glucosazone. To verify these results, a sample of the ethyl acetate fraction was hydrolyzed and analyzed for glucose. The samples (5 mg.) were sealed in small glass ampoules containing 1 ml. of 2% aqueous HCl and were heated in a steam bath for 0.5 hr. The hydrolyzates were analyzed for glucose by paper chromatography using ethyl acetate–acetic acid–water (9:2:2) (*25*) as the developer and *p*-anisidine hydrochloride in butanol (*26*) as the spray reagent. The amount of glucose in the hydrolyzate was estimated visually by comparing the area and depth of color of the glucose spots from the hydrolyzates with spots of known glucose concentration run on the same sheet. This method of estimation is probably accurate to ±5% (*27, 28*).

Hydrolyses run on the ethyl acetate fraction obtained by Pigman and the one obtained in this work yielded 20 ±5% glucose. This value is one half of that reported previously. Pigman used the Munson-Walker method of estimating reducing sugars. Since this method is sensitive to all types of reducing materials, the phenolic reducing materials present in the hydrolyzate probably caused the high result.

Isolation of the Leucoanthocyanin

The first attempts to isolate the leucoanthocyanin were made by using chromatography columns of unwashed Magnesol according to the procedure of Ice and Wender (*29*). This method proved unsuccessful, since the fraction from the column containing the leucoanthocyanin also contained fairly large amounts of dissolved inorganic materials which were difficult to remove. Furthermore, this fraction also contained several other compounds which could not be separated from the leucoanthocyanin. These experiments did indicate, however, that the leucoanthocyanin constituted no more than 10% of the ethyl acetate fraction.

The next method of isolation tried was countercurrent distribution using an amyl alcohol and water system. One gram of the ethyl acetate fraction dissolved in 20 ml. of water was extracted with 20 ml. of amyl alcohol. The upper layer was then extracted twice more with water so that there were three aqueous solutions, each of which was extracted five times with amyl alcohol in countercurrent fashion. The yields from the water solutions were: first solution 7.8%; second solution 2.6%; third solution 1.7% giving a total of 12.2% of the starting material. Two-dimensional chromatograms of the water solutions showed spots for the leucoanthocyanin and two other unknown compounds. Chromatograms of the amyl alcohol solutions showed that none of the leuco anthocyanin had been extracted by this solvent.

A nearly pure leucoanthocyanin was precipitated as a neutral lead acetate complex from an aqueous solution of the water-soluble fraction from the countercurrent distribution. A saturated solution of neutral lead acetate was added to a dilute aqueous solution of the water-soluble fraction until no further precipitation was observed. The solution was centrifuged, the resulting precipitate washed several times with water, and acidified with a few drops of acetic acid. Hydrogen sulphide was passed into the acidified solution until lead sulphide precipitation was complete. The lead sulphide was removed by centrifugation and the clarified supernatant solution extracted with ethyl acetate. A paper chromatogram of the ethyl acetate solution showed a faint spot for *d*-catechin and a strong spot for the leucoanthocyanin.

The latter was purified further by dissolving it in a minimum of methanol and precipitating it into ethyl ether (*11*). The off-white precipitate was collected by centrifuging, dried, and was found to be chromatographically pure. The yield of purified leucoanthocyanin was 0.7% based on the ethyl acetate fraction.

Characterization of the Leucoanthocyanin

The isolated leucoanthocyanin was treated with 95% ethanol 0.4 *N* in hydrochloric acid for 40 min. on a steam bath. The resulting red solution was concentrated *in vacuo* and chromatographed using acetic acid-concentrated hydrochloric acid-water (30:3:10) (*30*) and 90% formic acid-3 *N* HCl (1:1) (*31*) as the developers. The R_f values were 0.50 and 0.22, respectively, corresponding exactly to those reported by Roux (*31*) for cyanidin. The chromatograms also showed trailing streaks of "phlobaphene" material.

The Robinsons' (*4*) qualitative color tests were applied to the anthocyanidin. An amyl alcohol solution of the anthocyanidin was placed over 1% HCl and sodium acetate was added followed by a few drops of ferric chloride solution. The amyl alcohol layer turned blue indicating that cyanidin was present.

Strong confirming evidence for the identity of the anthocyanidin was obtained from spectral measurements. The wavelength of maximum absorption of a solution of the anthocyanidin and the shift in this maximum caused by complexing the anthocyanidin with aluminum chloride is quite diagnostic (*32, 33*). However, since the aluminum complex of cyanidin cannot be formed in acid solution (*33*), quite an experimental problem presented itself, because the leucoanthocyanin must be treated with acid to form the anthocyanidin. Several attempts to neutralize the acid in the ethanol solution led to the destruction of the anthocyanidin. Finally, by keeping the ethanol solution for several days in a vacuum desiccator over calcium oxide, its acidity was reduced to the point at which the aluminum complex could be formed. The absorption measurements were made using the solvent systems suggested by Geissman (*33*). Table II lists the wavelength of maximum absorption of the various solutions of the anthocyanidin and compares these values with those reported for cyanidin. The spectral evidence coupled with chromatographic evidence and the Robinsons' test established the anthocyanidin as

Table II. Absorption Maxima for the Anthocyanidin

Solvent	*Anthocyanidin from leucoanthocyanin, mμ*	*Cyanidin, mμ*
Absolute ethanol	545	546 (*32*)
50% ethanol	538	534 (*33*)
50% ethanol-$AlCl_3$[a]	568	567 (*33*)
50% ethanol-HCl-$AlCl_3$[b]	538	534 (*33*)

[a] Two drops of 2% alcoholic $AlCl_3$ added to the 3-ml. spectrophotometer cell containing the anthocyanidin in 50% ethanol.
[b] One drop of 0.5 *N* alcoholic HCl and two drops of 2% alcoholic $AlCl_3$ added to the 3-ml. spectrophotometer cell containing the anthocyanidin in 50% ethanol.

cyanidin, an identification which fixed the position of the aromatic hydroxyl groups of the leucoanthocyanin.

The identification of cyanidin and the chromatographic data indicating that this compound was a monomeric leucoanthocyanin suggested that the leucoanthocyanin might be the same as Swain's (*34*) synthetic "leucocyanidin" (3′,4′,5,7-tetrahydroxy-flavan-3,4-diol). Therefore, this compound was synthesized. A 100-mg. sample of dihydroquercetin was dissolved in 3 ml. of methanol and added to 5 ml. of aqueous 0.1 *N* NaOH containing 5 mg. of sodium borohydride. The reaction was allowed to continue for 1.5 hr. at room temperature whereupon the mixture was acidified and the methanol removed by distillation under reduced pressure. The aqueous solution was extracted with ethyl acetate and the extract taken to dryness *in vacuo*. The residue was dissolved in methanol and precipitated into ethyl ether. Three precipitations were necessary to remove the impurities. The yield of purified product was 22% of the theoretical yield.

To check the identity of the product, it was heated in ethanol in 0.4 *N* hydrochloric acid and treated in the same manner as the isolated leucoanthocyanin. The chromatographic and spectral behavior of the acid-treated synthetic product were the same as those of cyanidin.

Both the synthetic and isolated leucoanthocyanins had the same physical appearance when freshly precipitated and both turned dark brown upon standing. Paper chromatograms of the two materials yielded similar results.

Ultraviolet spectra were determined for both compounds in purified 95% ethanol with a Beckman DU spectrophotometer. The spectra, depicted in Fig. 1, are very similar except for the 220 to 235 mμ range where the isolated material shows slightly higher

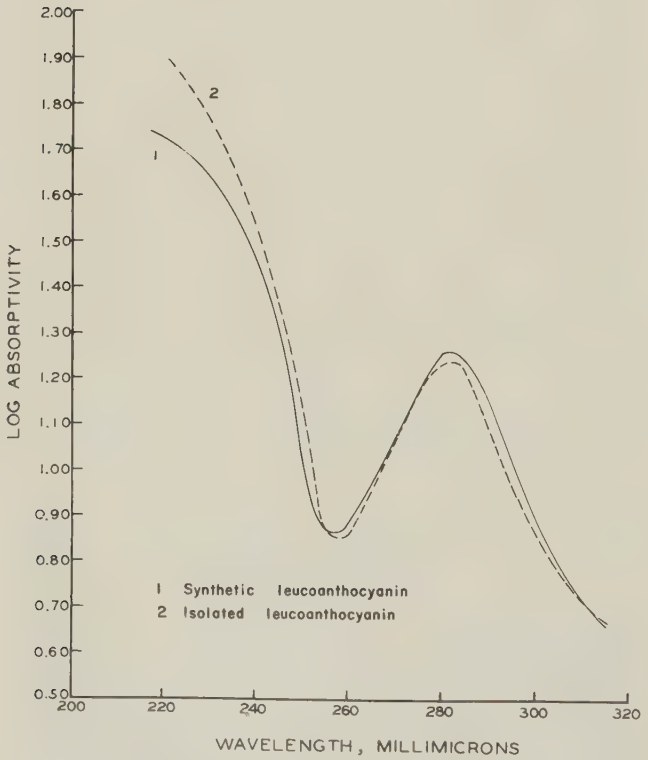

Fig. 1. Ultraviolet spectra of the isolated and synthetic leucoanthocyanins

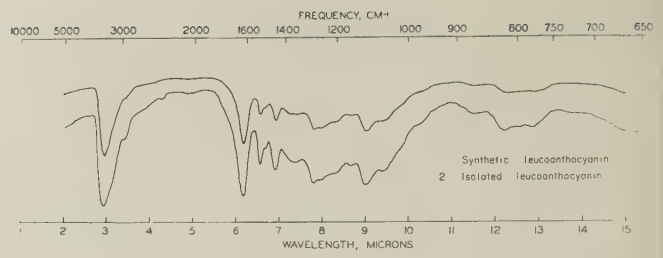
FREQUENCY, CM-1
10000 5000 3000 2000 1600 1400 1200 1000 900 800 750 700 650
Synthetic leucoanthocyanin
2 Isolated leucoanthocyanin
1 2 3 4 5 6 7 8 9 10 11 12 13 14 15
WAVELENGTH, MICRONS

Fig. 2. The infrared spectra of the isolated and synthetic leucoanthocyanins

absorption. It is doubtful that these differences are real, since the solvent and instrument cut-off are in this range so that the measurements are subject to error. The wavelength of maximum absorption for both compounds was at 282mμ and the minimum at 258 mμ. The absorptivities were 17.5 for the isolated leucoanthocyanin and 18.1 for the synthetic product.

The infrared spectra of the synthetic and isolated leucoanthocyanins were indentical as shown in Fig. 2. The spectra were determined with a Perkin Elmer Model 21 infrared spectrophotometer using the potassium bromide pellet method. The noncrystallinity of these compounds may be responsible for the broadening of the bands in the 650 to 1000 $cm.^{-1}$ region and the general lack of sharpness over the whole spectrum.

The Acetate

The low yield of leucoanthocyanin suggested that the acetate prepared by Pigman, *et al.* (*18*), was not a derivative of the leucoanthocyanin, but of some other compound. To investigate the nature of the acetate, an acetylation was made following the procedure of Pigman. One gram of the ethyl acetate fraction was dissolved in 7 ml. of pyridine, the solution cooled to 0°C., and 15 ml. of acetic anhydride at 0°C. were added. The reaction mixture was kept at 0°C. for 48 hr. and then poured into 80 ml. of ice and water. A sticky gum formed which powdered upon rubbing. The yield was 1.70 g. The crude product was crystallized once from methanol and crystals were obtained which melted at 153–154°C. and had a rotation of $-18.37 \pm 1.10°$ (0.025 g. per ml.) in acetone. A mixed melting point of this product and the acetate obtained by Pigman showed no depression. This crystalline product was recrystallized five more times from methanol. The last crystallization yielded long needles which melted at 161 to 163°C. and had a rotation of $-20.30 \pm 1.14°$ (0.022 g. per ml.) in acetone. These values corresponded very closely with those obtained by Andrews (*19*) for his stilbene glucoside acetate (m.p. 162 to 163°C. and rotation −22.6° in acetone) and indicated that the compounds were identical.

To verify the identity of the acetate obtained in this work, it was oxidized with potassium permanganate in acetone according to the procedure of King, *et al.* (*35*). A 48-mg. sample of the purified acetate (m.p. 161 to 163°C.) was dissolved in 10 ml. of acetone and 40 mg. of potassium permanganate in 20 ml. of acetone was added. The reaction was allowed to continue at room temperature for 16 hours, whereupon the residual permanganate was destroyed with a few crystals of sodium bisulfite. The manganese dioxide was filtered off, washed with hot water, and the wash water

and the acetone filtrate combined and evaporated until the acetone was completely removed. The solution was acidified and extracted with ethyl ether. The ether solution was taken to dryness, the residue suspended in dilute sodium hydroxide, and warmed until all the material had dissolved. The solution was then acidified and extracted with ether. Chromatograms of the ether extract run in benzene–acetic acid–water (2:2:1) (*36*) showed a spot for vanillic acid and an unknown spot at the origin.

The ether solution was taken to dryness and 10 ml. of 10% H_2SO_4 was added. The solution was refluxed for 0.5 hr., cooled, and extracted with ether. A chromatogram of the ether extract run in benzene–acetic acid water showed spots for vanillic acid and α-resorcylic acid (3,5-dihydroxybenzoic acid). Chromatograms of the products from an oxidation and sebsequent hydrolysis of the impure acetate (m.p. 153–154°C.) showed strong spots for vanillic acid, α-resorcylic acid, *p*-hydroxybenzoic acid, and protocatechuic acid (3,4-dihydroxybenzoic acid).

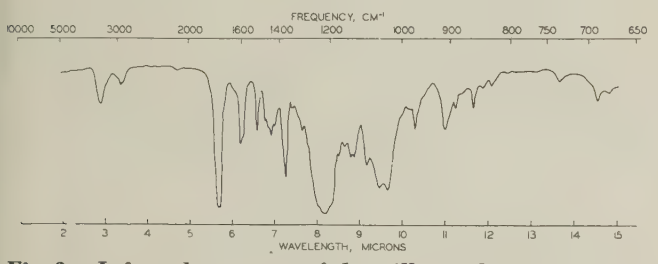

Fig. 3. Infrared spectrum of the stilbene glucoside acetate

A sample of the purified acetate analyzed for carbon, hydrogen, and methoxy] gave the following results: C, 59.0%; H, 5.54%; OCH_3, 4.40% (calculated for $C_{33}H_{36}O_{15}$: C, 58.9%; H, 5.36%; OCH_3, 4.62%). Andrews (*19*) reported an analysis: C, 58.9%; H, 5.5%; OCH_3, 4.5%. He obtained vanillic acid and α-resorcylic acid upon oxidation of his stilbene glucoside acetate.

An infrared spectrum of the stilbene glucoside acetate was determined and is shown in Fig. 3. The prominent absorption bands may be correlated as follows. The absorption at 1755 cm.$^{-1}$ is attributable to the ester carbonyl, while the absorption at 1195 to 1240 cm.$^{-1}$ is due to phenol acetates and aliphatic hydroxyl acetates. The doublet at 1595 to 1610 cm.$^{-1}$ is probably due to a C=C conjugated with the aromatic rings and aromatic absorption. The second aromatic band is at 1510 cm.$^{-1}$. The sharp band at 970 cm.$^{-1}$ may be due to the glucoside portion of the molecule or more probably to a trans-ethylenic configuration. Kuhn (*37*) has shown that methyl β-D-glucoside tetraacetate absorbs strongly at 910 cm.$^{-1}$ and that D-glucal-3,4,6-triacetate and glucose absorb strongly at 1370 cm.$^{-1}$. Therefore, the bands at 1370 and 910 cm.$^{-1}$ are due presumably to the glucoside portion of the molecule.

THE PROPANOL HYDROLYSIS TEST

Pigman, *et al.* (*18*), devised a semiquantitative test for the estimation of leucoanthocyanins, which was based on the spectrophotometric measurement of the anthocyanidin produced by acid treatment. The test results were reported as absorptivities since no method of standardization was available. They found the relation between the absorbance of the final solution and the initial concentration was not linear. To eliminate discrepancies due to this nonlinearity, they specified that a sample size be taken that would yield a final transmittance of 20 to 30%.

Table III. Standardization of the Propanol Hydrolysis Test

Concentration, mg./10 ml.	*Absorbance*	*Absorptivity*
2.84	1.560	5.51
1.12	0.859	7.65
0.56	0.495	8.91
0.28	0.307	10.79
0.12	0.133	11.60
0.06	0.080	14.36

The synthetic leucoanthocyanin appeared to be a suitable material for the standardization of this test. Accordingly, a series of samples of different concentrations were used for standardization. The results, recorded in Table III, agree with the previous work in that the absorbance of the final solution is not a linear function of initial concentration. A calculation of the concentration of anthocyanidin, based on the extinction coefficient of cyanidin, showed that the conversion to anthocyanidin was only 7 to 20% of theoretical with conversion increasing with decreasing initial concentration.

The propanol hydrolysis test was applied to the ethyl acetate fraction over a range of concentrations. The results of the tests are given in Table IV. The percentage leucoanthocyanin was calculated from these data using the standard to determine the concentration of true leucoanthocyanin. In the concentration range studied, the calculated percentage of leucoanthocyanin varied from 9.1 to 11.1%. Calculating the percentage leucoanthocyanin from the absorption data reported by Pigman, *et al.* (*18*), an average value of 30% was obtained. Evidently, Pigman's preparation was richer in leucoanthocyanin than was the preparation obtained in the present work. When using the standard, the measurements need not be limited to the 20 to 30% transmittance range, since consistent results can be obtained over a transmittance range of 20 to 80%.

The values given by this test may be somewhat high due to the absorption of extraneous materials. Corrections can be made for materials which absorb prior to acid treatment by making absorption measurements before acid treatment. In this work the absorption of the original solutions was negligible. However, nonleucoanthocyanin materials which absorb only after acid treatment would produce an error which cannot be corrected.

Caution should be exercised in using the standard established in this work for the calculation of the percentage leucoanthocyanin in other extracts. If the relationship between concentration and absorbance

Table IV. Propanol Hydrolysis Test on the Ethyl Acetate Fraction

Concentration, mg./10 ml.	*Absorbance*	*Absorptivity*	*Leucoanthocyanin, %*
4.55	0.412	0.91	9.1
2.75	0.305	1.10	10.7
2.00	0.204	1.02	9.5
1.44	0.170	1.18	9.7
0.60	0.064	1.07	10.0
0.36	0.045	1.25	11.1

is different or if the percentage conversion varies, erroneous results will be obtained by using this standard. Also, leucoanthocyanins which produce anthocyanidins other than cyanidin may require a different standard. Further work would be required to fully evaluate this method of testing.

DISCUSSION

The 0.7% yield of isolated leucoanthocyanin probably represents a minimal figure, since losses during the isolation undoubtedly reduced the yield. Also there was probably additional loss due to decomposition of the leucoanthocyanin. Apparently, the leucoanthocyanins used in this work were unstable, since within a few days they became discolored and their solubility decreased. Due to this instability, an accurate estimation of the leucoanthocyanin originally present in the ethyl acetate fraction is quite difficult. However, the estimated value of 10% leucoanthocyanin as determined by the propanol hydrolysis test and supported by countercurrent distribution and column chromatography yields is a reasonable figure. Since this figure represents the value for only three trees, it cannot be taken as a true representation of the amount present in the inner bark of black spruce in general.

The structure of the leucoanthocyanin has been quite well established as 3′,4′,5,7-tetrahydroxy-flavan-3,4-diol. This structure agrees with the current concept of the leucoanthocyanins, since the flavan-3,4-diol has been generally accepted as one of the basic structural types for the leucoanthocyanins (*8–10*). Treatment of this compound with hot dilute acid produced the expected results, i.e., cyanidin and a water-insoluble phobaphenelike precipitate. These results coincide with the data of Swain (*34*) for his synthetic "leucocyanidin" and also with the results obtained by King and Bottomley (*8*), although their anthocyanidin differed from cyanidin because of a different hydroxylation pattern.

During the isolation and characterization of the stilbene glucoside acetate, data were obtained which indicated the presence of at least one other compound. This was first indicated in the oxidation experiments, when four substituted benzoic acids were detected among the oxidation products. The vanillic acid and α-resorcylic acid were derived from Andrews' stilbene glucoside; the source of the *p*-hydroxybenzoic acid and the protocatechuic acid remains unknown. Since these two acids could not be detected in the original ethyl acetate fraction, they probably resulted from some compound or compounds with linkages susceptible to oxidation under the conditions used in this work. Since the protocatechuic acid could not be detected among the oxidation products until after they had been hydrolyzed, it appears that one of its hydroxyls was combined in some manner, probably as a glycoside. The sugar residue was doubtless glucose, as it was the only one detectable among the hydrolysis products of the ethyl acetate fraction.

The leucoanthocyanin has been shown to be but a relatively small part of the ethyl acetate fraction and that it contains neither a methoxyl group nor a glucose residue. Since the stilbene glucoside contains both a methoxyl and a glucose residue and appears to represent a large portion of this fraction, it seems probable that the data furnished by Pigman, *et al.* (*18*) are more representative of the stilbene glucoside than of the leucoanthocyanin. The ethyl acetate fraction irrespective of whether isolated by Pigman or in the present work has properties apparently identical with those of Andrews' stilbene glucoside. Both materials had about the same rotation. The acetate as prepared by Pigman had properties similar to the acetate of Andrews' stilbene, and has now been shown to contain this compound. The elemental and functional group analyses of the Pigman acetate were almost the same as those for Andrews' stilbene. Thus, it seems probable that the Pigman acetate was an impure preparation of Andrews' stilbene glucoside.

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Physical Properties of Bleached High Yield Pulps

NOEL N. COE and H. LANCE CROSBY

A high yield semichemical pulp similar to cold soda process pulp was prepared and separate portions were bleached by a total of five different bleaching agents. The bleached pulps were analyzed chemically and subjected to beater evaluations in order to compare the physical properties of the pulps as influenced by the bleaching conditions and agents. It was found that sodium hydrosulfite, peroxide, sodium hypochlorite, and chlorine dioxide have a generally similar action. At a given freeness, the strength properties and ease of beating are improved, the apparent density is increased, and the opacity is decreased compared to unbleached pulp. Sodium hydrosulphite appears to have the least effect of the four and sodium hypochlorite generally the most. A two-stage chlorine-sodium hydroxide treatment had a very much greater effect. It resulted in a tremendously increased apparent density and pulp strength, as compared to the other bleaches. The loss in opacity also became extreme after a short beating time. This bleach was also quite different from the others in that no real brightening was affected, but merely a purification.

It is well known that different bleaching agents have different effects on pulps. Much work has been done in this field and especially in the bleaching of chemical pulps. However, the reactions of the various well-known bleaches with semichemical pulps have been explored to a lesser extent, especially in the case of the higher yield semichemical pulps, typical of which is the "cold soda" process pulp.

These pulps respond quite differently from either the chemical or groundwood pulps. The former have small amounts of fairly inert material that must be removed or altered in order to produce a bleached pulp. In the case of the latter there are large amounts of relatively reactive materials that must be treated. However, in the case of the high yield semichemical pulps there are substantial amounts of intermediately reactive materials. Bleaching them provides a very interesting problem, because the slight differences in the action of the different bleaching agents show up to a considerable degree.

PULPING PROCESS

A typical high yield semichemical pulp was made according to the flow sheet shown in Figure 1. The raw material was unbarked black birch chips. The Kollermill® machine was used both as impregnating device and then later with a refining attachment added as a refining device in order to assure the highest yield of pulp and the least production of fiber debris. The system was closed by the return of white water from the decker to the screen for dilution. The only waste produced was the cleaner reject material.

This process results in producing a pulp that is substantially free of fiber bundles, yet the integrity of the primary cell wall is well preserved. There is then a rather tender fiber still protected by a tough lignified sheath. The freeness was close to 500 ml., Canadian Standard, and the fiber-to-fiber bonding strength quite low.

The problem was to compare different bleaching agents that would not only brighten the pulp from the initial low brightness value of 30 but would also so modify the fiber sheaths as to produce the highest sheet strength.

BLEACHING CONDITIONS

Five portions were taken from one batch of an experimental pulp and were treated with the following five bleaches: sodium hydrosulphite, peroxide, sodium hypochlorite, chlorine dioxide, and finally, a two-stage chlorination-caustic extraction sequence. The conditions and results of bleaching portions of the pulp are summarized in Table I. The batch size was on the order of 600 g. (dry weight), except in the case of the chlorine-caustic extraction bleach where an 800-g. portion was taken in order to allow for the expected amount of shrinkage.

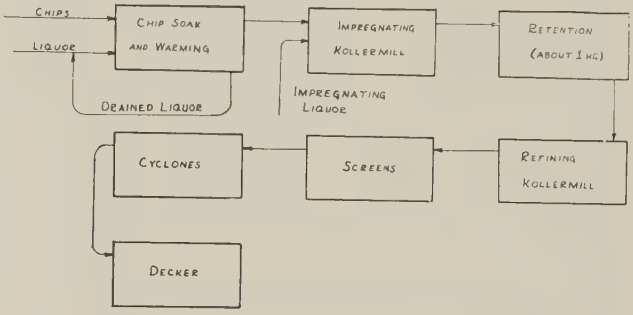

Fig. 1. Flow sheet for pulp production

The bleaching conditions chosen for the test were those generally accepted as being close to optimum. An exhaustive investigation of optimum conditions for all these bleaches was not undertaken at this time. Furthermore, it was felt that it would then be necessary to study a whole series of optimum conditions since optimum brightness, for example, might not result from the same conditions producing optimum mullen.

While initial pH was adjusted as shown in Table I, neither an acid sour nor even a wash was applied before bleaching the pulp, since this would alter the physical characteristics. This also was largely responsible for the initial low brightness of the pulp which was 30 at pH 7 and 29 at pH 5.

The results shown in Table I indicate that under the conditions of the test, sodium hypochlorite produces the greatest brightness increase and yellowness reduction at the least cost. Chlorine dioxide is next, followed

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Table I. Bleaching Data

Bleaching Agent	%[a]	Other chemicals	%[a]	Bleach: Concn., %	Bleach: Temp., °C	Bleach: Time, hr.	pH: Initial	pH: Final	Brightness, gain, pts.	Yellowness, reduction, pts.	Steam and chemical cost $/pt. brightness gain ton[d]	Permanganate no.[e]
Sodium hydrosulfite	1	Sulfur dioxide as needed for initial pH adjustment sodium tripolyphosphate	0.5	4	66	0.75	6.0[d]	6.5	7	4	1.28	132
Hydrogen peroxide	2	Epsom salts Sodium silicate Sodium hydroxide	0.05 5.0 1.3	12	85	1.75	10.5	10.3	17	3	1.30	127
Chlorine dioxide	10[c]	None	...	12	62	4	6.5	5.8	18	6	0.98	121
Sodium hypochlorite	10[c]	Sodium hydroxide	1.3	12	32	1.25	10.4	9.2	30	7	0.61	118
Chlorine	16	None	...	3.5	23	1.5	1.5	1.6	3	5	6.03	81
Sodium hydroxide	5.7	None	...	12	66	1.5	11.2	9.4				

(a) Basis: ovendry pulp.
(b) Before addition of hydrosulfite and polyphosfate.
(c) As available chlorine.
(d) Cost data (cts./lb.): sodium hydrosulfite, 24; sodium tripolyphosfate, 10; sodium peroxide, 20.5; hydrogen peroxide (100%), 51.4; epsom salts, 5.0; sodium silicate, 1.5; chlorine dioxide, 22.4; sodium hypochlorite, 8.5; sodium hydroxide, 3.8; chlorine, 4.0; steam, 0.07.
(e) Permanganate number of unbleached pulp, ovendry pulp, 135.

by peroxide. Sodium hydrosulfite, as might be expected, did not increase the brightness a great deal. The chlorine-caustic extraction two-stage treatment probably did not actually have any brightening action at all on the remaining pulp, but merely dissolved out the darkest components. The peroxide bleach would probably have brightened up much better with an initial acid sour (*1*).

BLEACHING SHRINKAGE

The raw and bleached pulps were analyzed for alpha, beta, and gamma-cellulose using the procedure set forth in TAPPI Standard T 429 m-48 while taking into consideration the work of Launer (*2*) which covers the analysis of groundwood type pulps.

Lignin was determined separately by TAPPI Standard T 222 m-54. This value was then subtracted from the alpha-cellulose value of T 429 since, according to Launer, the lignin is effectively all in the alpha-cellulose portion. The reported alpha-cellulose is then this net figure. The beta and gamma-cellulose figures remain as determined in T 429.

Yields were also determined on the bleaches.

These results are shown in Fig. 2. The length of the bars illustrates the values on the raw pulp basis.

It is considered significant that the total alpha, beta, and gamma-cellulose of the sodium hydrosulfite, peroxide, and sodium hypochlorite bleaches is close to 73% of the raw pulp in each case, whereas the lignin is successively diminished.

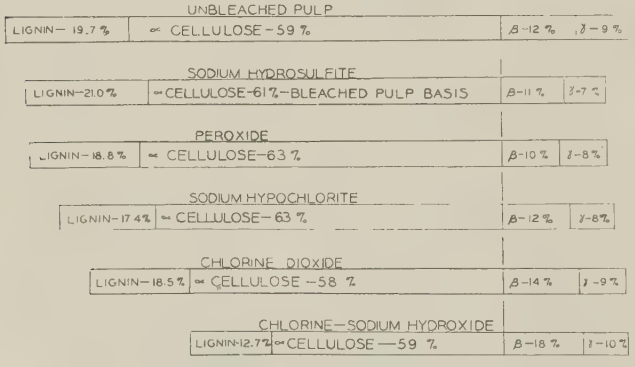

Fig. 2. Bleaching yields and pulp analyses

In the case of the chlorine dioxide bleach, the lignin is reduced to the same value as in the sodium hypochlorite bleach but the alpha-cellulose is further reduced, part of it apparently showing up as beta, and gamma-cellulose.

In the chlorine bleach followed by caustic extraction, the lignin is reduced to 45% of the amount in the raw pulp, but the alpha-cellulose is at the same time reduced to 70% of that in the raw pulp. Again, part of the alpha-cellulose is probably degraded.

In other work done with unbleached pulp of this type, it has appeared that approximately 8–10% of the pulp can be removed as colloidal matter during dewatering and washing. This is offered as an explanation of the approximately 8% shrinkage in the sodium hydrosulfite and peroxide bleaches. It is postulated that this is solely a mechanical loss that occurred while washing out the excess bleach.

(NOTE: The fiber classification does not reflect this loss in fines, since all the pulp, including the consistency determination, was caught on coarse filter papers through which the colloidal matter can pass, whereas, the yield determinations were made by evaporating wet pulp samples.)

Furthermore, other data have indicated that in high yield semi-chemical pulps, not all the lignin is acid insoluble and that the value obtained by the 72% sulfuric acid method will be low. It is believed that the actual amount of lignin in the unbleached pulp is somewhat greater than the 19.7% shown. Also the retention of the pulp at an elevated temperature during hydrosulfite bleaching could cause the lignin to react so as to become less acid soluble. If a substantial part of the total lignin is now removed during washing (and tests have indicated that about half of the filtrate solids are lignin) it is possible for the amount of lignin remaining in the pulp after the sodium hydrosulfite bleach to be coincidentally quite close to that in the raw pulp; i.e., 19.3 versus 19.7%.

FREENESS IN BEATING

The raw and bleached pulps were subjected to beater evaluations, following TAPPI Standard Method T 200 m-45. Figure 3 shows the change in freeness during beating. It is quite noteworthy that while all the

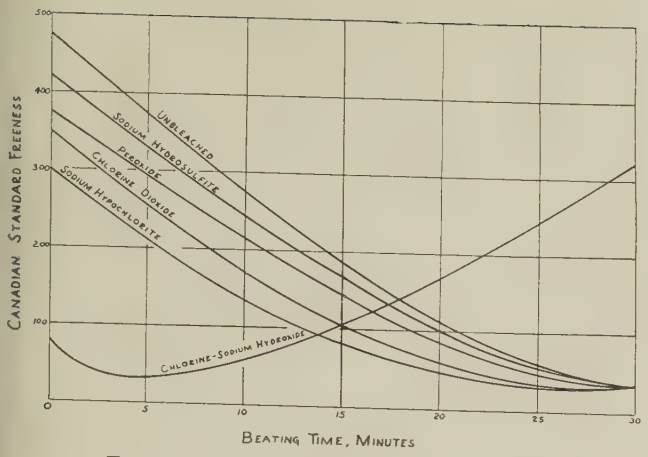

Fig. 3. Loss of freeness during beating

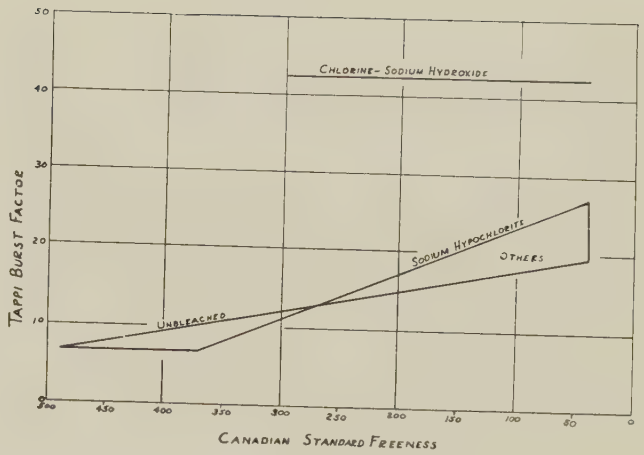

Fig. 4. Mullen development during beating

pulps would be considered "easy beating" the relative positions of the curves do not follow the hemi-cellulose content or the unbeaten fiber classification as shown in Table II as would be expected. For example, the fiber classification of the chlorine-caustic extraction pulp might indicate that it would have a freeness close to the unbleached pulp which was not the case. Instead, the lignin content or the amount of intact fiber sheath it reflects is apparently the determining factor.

The shape of the chlorine-sodium hydroxide curve after the first 5 min. is explained not as a true increase in drainage rate but only as an increase in the value determined by the freeness test. It was quite apparent during the making of the handsheets that actually the drainage rate dropped throughout the run.

It is postulated that the order of the freenesses at time "zero" is governed by a sensitivity to "hydration" as influenced by the bleach. All the pulps received about the same handling, with the exception of the chlorine-sodium hydroxide, which, naturally, received double handling because of its two stages. These tests along with a variety of others that have been run in the laboratory indicate that the drop in freeness during bleaching is substantially independent of the amount of bleaching agent used and, instead, may be the result of the physical work done on the fiber while the pulp is sensitized by the bleach.

BURST, TEAR, AND TENSILE

The TAPPI burst factor, tear factor, and tensile

Table II. Fiber Classification

Mesh	On 14	On 30	On 50	On 100	Through 100
Unbeaten					
Unbleached	0	7	39	14	40
Sodium hydrosulphite	0	5	34	20	41
Peroxide	0	6	29	16	49
Chlorine dioxide	0	4	36	23	37
Sodium hypochlorite	0	5	34	20	41
Chlorine-sodium hydroxide	0	5	41	20	34
Beaten 30 Minutes					
Unbleached	0.2	2.6	27	17	53
Sodium hydrosulphite	0	2.9	29	17	51
Peroxide	0.4	1.5	29	18	51
Chlorine dioxide	0	3.1	31	19	48
Sodium hypochlorite	0	2.2	29	18	51
Chlorine-sodium hydroxide	[a]				

[a] Blinded screens and could not be run.

breaking length were run according to TAPPI Standard T 220 m-53. They are plotted against freeness in Figs. 4, 5, and 6.

The sodium hydrosulfite bleach developed no strength over that in the unbleached pulp. The peroxide, chlorine dioxide, and sodium hypochlorite bleaches

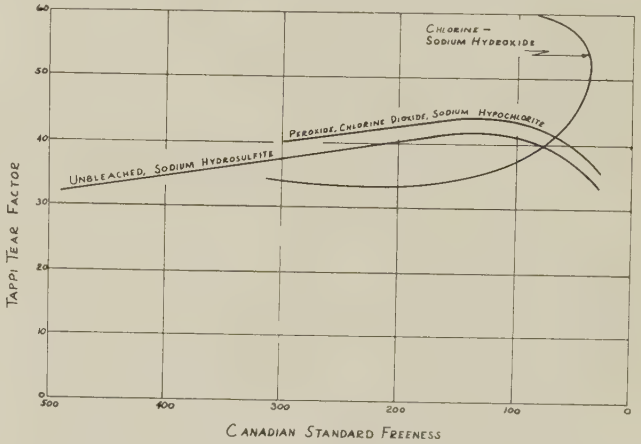

Fig. 5. Tear development during beating

apparently conditioned the pulp to develop some additional strength which shows up after some beating. The burst factor graph is especially interesting in that the lines apparently cross at about 275 freeness.

The chlorine-sodium hydroxide has a far higher burst and tensile than the others and the tear strength also starts off quite high but soon drops as a result of beating.

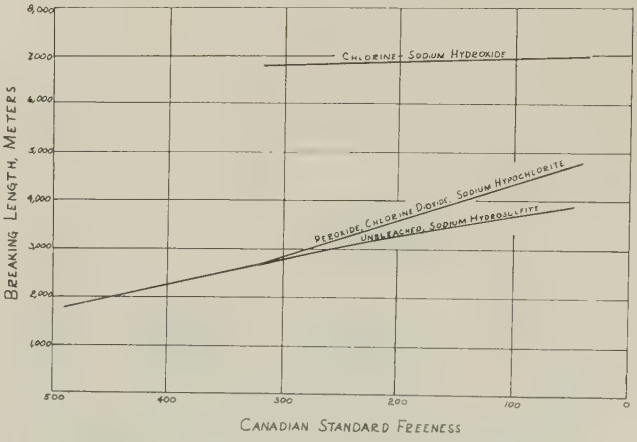

Fig. 6. Tensile strength during beating

ZERO SPAN TENSILE

Figure 7 displays a rather confused but very interesting set of lines representing a plot of zero span breaking length versus freeness developed during beating.

The unbleached pulp apparently develops some strength during beating, possibly by making a more uniform pulp which forms a smoother sheet that fills the jaws of the grips better and results in a higher strength. However, after beating, the fibers are apparently damaged and the strength falls off.

The sodium hydrosulfite follows the same development although there appears to be another factor involved that suppresses the development of a maximum. The chlorine dioxide bleach also shows the same inter-

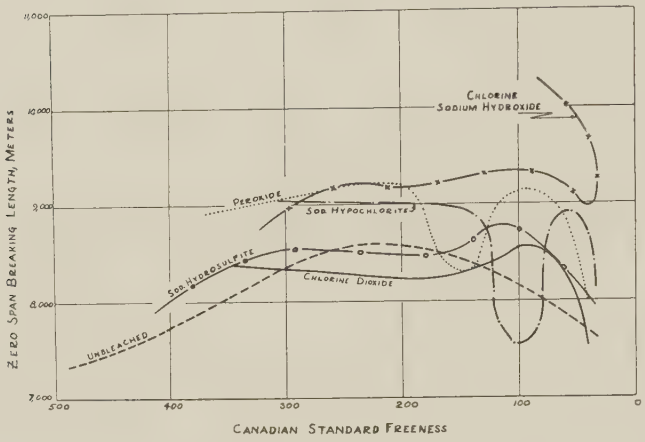

Fig. 7. Zero span during beating

action with a slight dip before the maximum. The low position of the chlorine dioxide line is undoubtedly a reflection of the reduced alpha-cellulose content. In the case of the sodium hypochlorite and peroxide bleaches this dip effect is very much pronounced.

As usual the chlorine-sodium hydroxide starts at a high value, then as the freeness goes through a minimum it follows the same pattern dipping before rising and finally tapers off to a lower value toward the conclusion of the beating.

While it was first thought that there might be something in the testing procedure that resulted in such a confusion of lines, upon investigation it was established that the test values of the octuplicate samples were too close together and the pattern too well repeated to be the result of anything but a response to the interplay of factors. Furthermore the variations were much too great to be considered deviations from a smooth curve. This will bear further investigation.

APPARENT DENSITY AND OPACITY

The plot of apparent density versus freeness during beating (Fig. 8) is interesting in that it shows an increased sensitivity to beating after bleaching. For while the relationship between apparent density and freeness is essentially linear in the case of the unbleached pulp, the bleached pulps have a marked degree of curvature.

The high level of apparent density of the peroxide bleach is reflected in the fiber classification results in Table II where a reduced amount of material retained on the 50-mesh screen and large amount of material passing 100 mesh is found. It is worth noting, however, that this does not result in an improvement in strength.

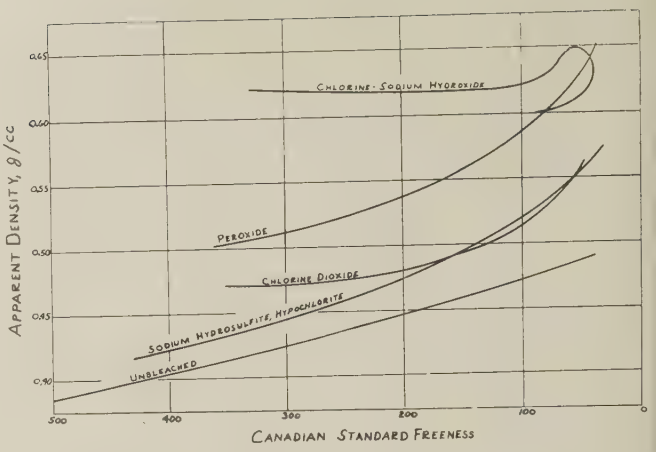

Fig. 8. Apparent density during beating

In the same way, the fiber classification would lead one to expect that the chlorine-sodium hydroxide bleach would have the least apparent density, but, perversely the opposite is true.

The shape of the chlorine-sodium hydroxide curve is interesting in that after increasing in apparent density very rapidly, it goes through a maximum, and then after the freeness curve reverses, it drops and levels off at a density only slightly greater than the unbeaten pulp.

This chlorine-sodium hydroxide is also a case by itself in Fig. 9, the other bleaches show very little drop in opacity as a result of beating, while following the same order for brightness gain. The chlorine-sodium hydroxide treatment with no brightness gain has an initial opacity equivalent to the brightest and then drops very fast until eventually it levels off.

FIBER CLASSIFICATION

The fiber classification results with the Clark classifier have been referred to previously. Classifications were made on the unbeaten pulp as well as that which was beaten for 30 min. In the case of the chlorine-sodium hydroxide bleach, the drainage rate was so low that the water could not be made to flow through the screens at a sufficient rate to keep from overflowing the machine. It was thought that any change in procedure to circumvent this would so affect the results as to render them not worth while.

The small fines content of the unbeaten chlorine-sodium hydroxide pulp is probably due to dissolution of the particles having the greatest surface area and presumably also the greatest amount of lignin.

Otherwise, in spite of the variations in classifications of the unbeaten pulp, the beaten pulps all have about the same classification.

CONCLUSIONS

Sodium hydrosulphite bleaching of very high yield pulp does not appear to effect any substantial change in the major components of the pulp other than a small overall brightening and possibly a decrease in the acid solubility of the lignin. The fiber bonding strength is unchanged as compared to the unbleached pulp in burst, tear, and tensile. However, the drastic conditions of the bleach do appear to influence a breakdown of the 30 and 50-mesh fibers into 100-mesh material and this results in a slight increase in apparent density and zero

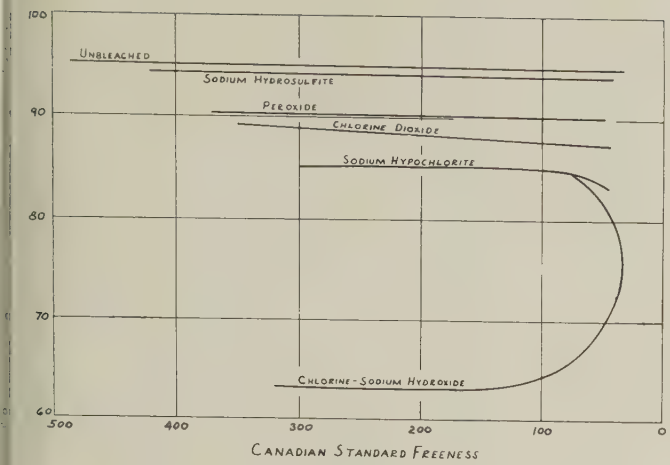

Fig. 9. Opacity changes during beating

span tensile and a slight decrease in opacity.

The peroxide bleach appears to attack the lignin, reducing the amount of it and producing a large amount of fines. Thus, the apparent density and zero span are high. The slightly higher fiber bonding strength shown in the tensile curves is probably also influenced by the bleach.

Chlorine dioxide dissolves more of the lignin than peroxide and also reduces the alpha-cellulose. As a result of these two factors, more of this pulp is found on the 100-mesh screen than on any other. Thus, the apparent density is high but not as high as in the peroxide bleach where so many fines were produced. The low zero span also reflects the reduced alpha-cellulose content. Of the two factors, the high apparent density appears to exert a greater influence than the low zero span and improved strengths result as compared with the unbleached pulp.

The sodium hypochlorite reacts in a slightly different manner again. The chemical attack removes about as much lignin as does the chlorine dioxide, while the cellulose is less attacked. The result is a high brightness and good strength. While the fiber classification and therefore apparent density are the same as for the sodium hydrosulfite, the zero span, burst, tear, and tensile are higher. This means that the fibers themselves are stronger and are more strongly bonded to each other.

The chlorine-sodium hydroxide is obviously a completely different kind of bleach. Thirty per cent of the alpha-cellulose and 55% of the lignin have been removed; furthermore, the fines passing 100-mesh are very low. Instead of this resulting in a high brightness and freeness, the former is substantially unchanged and the latter has been greatly reduced. This purification of the pulp has increased its burst, unbeaten tear, tensile, unbeaten zero span tensile, and apparent density tremendously. It is a strong but not a tough pulp and a little beating drops the tear and zero span very rapidly. The drop in fiber strength, as shown in the zero span curve, which drops the greatest amount of all the bleaches during 30 min. of beating, apparently balances the increase in fiber bonding due to hydration during beating because burst and tensile remain close to constant.

Thus, it is evident that the situation is complex and the interactions of the variables are pronounced. The overall effects are clearly shown; however, a statistical treatment of many more data than have been accumulated would be required to determine the actual significance of the variations found.

This work shows, however, that the different bleaching agents have quite different effects on the physical and chemical properties of a high yield semichemical pulp, pointing to the possibilities of "tailoring" a bleaching sequence to a specific end use.

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Acetylation of Blends of Wood Pulp and Cotton Linters

ARNOLD J. ROSENTHAL and B. B. WHITE

The rates of acetylation of acetic acid-swollen wood pulp/-cotton linters mixtures were determined by a reproducible high dilution acetylation procedure. In general, in sulfuric acid catalyzed acetylation reactions wood pulp reacted more rapidly than cotton linters. As expected, when the sulfuric acid catalyst was incorporated in the acetic acid-acetic anhydride acetylation mixture, wood pulp/cotton linters blends reacted at a rate intermediate between the rates for the individual cellulose sources. However, when the sulfuric acid was added to the swollen cellulose prior to the addition of acetic anhydride, wood pulp/cotton linters blends reacted more slowly than the pure components. By applying the sulphuric acid solution to preswollen wood pulp and cotton linters in separate containers, and then blending for acetylation, a rate intermediate between that for pure wood pulp and pure cotton linters was again observed. The phenomenon has been attributed to the preferential adsorption of the sulfuric acid by the wood pulp component of the cellulose blend. The results have implications in large scale reactions of blends of celluloses.

IN MANY commercial processes for the manufacture of cellulose acetate the cellulose is made to react with acetic anhydride in acetic acid solution using sulfuric acid as catalyst. The progress of the reaction is readily apparent because the cellulose is insoluble in the reaction medium whereas the acetylated product dissolves.

There are numerous practical reasons for desiring to measure the reactivity of a cellulose sample in the acety-

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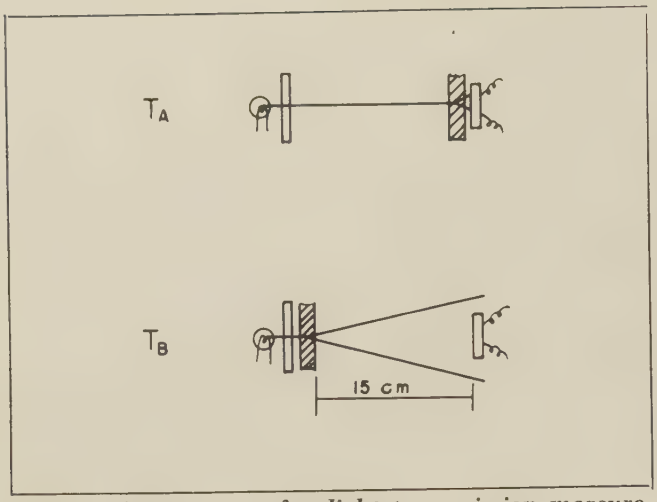

Fig. 1. Arrangement for light transmission measurements

lation reaction (*1*). For example, it would be useful to:

(a) Compare cellulose samples from different suppliers.
(b) Study the effects of pulp processing variables.
(c) Assess the effectiveness of cellulose swelling procedures.
(d) Study the chemical kinetics of the acetylation reaction.

To this end many investigators have reported on the feasibility of conducting acetylation tests. Borgin (*2*, *3*, *4*), Blume and co-workers (*5*), and Malm and co-workers (*6*) have made recent significant literature contributions in this field.

A procedure is described in detail in this paper for comparing cellulose reactivities which offers some interpretive advantages over procedures previously reported. Application of this acetylation procedure has disclosed an interesting phenomenon in the acetylation of blends of wood pulp and cotton linters celluloses.

EXPERIMENTAL

Acetylation Rate Procedure

The acetylation procedure consists of reacting a preswollen cellulose sample with acetic anhydride and sulphuric acid at relatively high dilution (~2% cellulose) in acetic acid. The reaction is conducted in a stirred reaction flask immersed in a constant temperature bath. At high cellulose dilution there is no difficulty in maintaining an isothermal reaction. The course of the reaction is followed by measuring the light transmission of aliquots removed from the reaction flask from time to time.

The light transmission measurements are made in a Lumertron Model 402-E photoelectric colorimeter in a manner so that the effect of any color absorption in the reaction mixture is minimized and the scattering effect of undissolved (presumably unacetylated) cellulose particles is exaggerated. Figure 1 illustrates that when the sample holder is in position A, adjacent to the photocell, the loss in transmission due to scattering by undissolved particles is minimized; whereas when the sample holder is in position B, farther from the photocell, the loss in transmission due to scattering is exaggerated. Reduction in transmission due to color of the solution should be identical in both positions. The ratio of transmission in position A to transmission in position B, i.e., T_A/T_B, is indicative of the concentration of undissolved cellulose in suspension, and independent of the solution color.

Table I. Optical Calibration Data
Cotton linters no. 5

Suspension, %	T_A	T_B	T_A/T_B
0.0	100	98.7	1.02
0.1	88.6	50.9	1.7
0.2	79.3	26.9	2.9
0.3	70.9	16.8	4.1
0.4	65.1	10.3	6.2
0.6	52.3	3.5	14.6
0.8	44.3	1.8	24.1
1.0	36.4	1.2	29.7

By making T_A and T_B light transmission measurements on suspensions containing known concentrations of acid-degraded cellulose particles it is possible to establish the relationship of T_A/T_B ratio to concentration. One per cent slurries of wood pulp and cotton linters celluloses in 2.5% sulphuric acid/97.5% acetic acid were degraded for periods from 16 to 64 hr. Aliquots of these suspensions were diluted with acetic acid (containing 2.0% of high clarity cellulose acetate to slow the settling of the suspension) to provide known suspension concentrations between 0.05% and 1.0%. The measurements were made with 620mμ light. A typical set of data for a cotton linters suspension is shown in Table I. The averaged results for five cotton linters samples and for two wood pulp samples are shown in Fig. 2. This figure shows that Log (T_A/T_B) versus concentration produces straight line plots over the range 0 to 0.4% concentration, but that the relationship differs for wood pulp and cotton linters. Thus, over this range the Beer-Lambert relationship is applicable.

$$\text{Log}\,\frac{T_A}{T_B} = kc$$

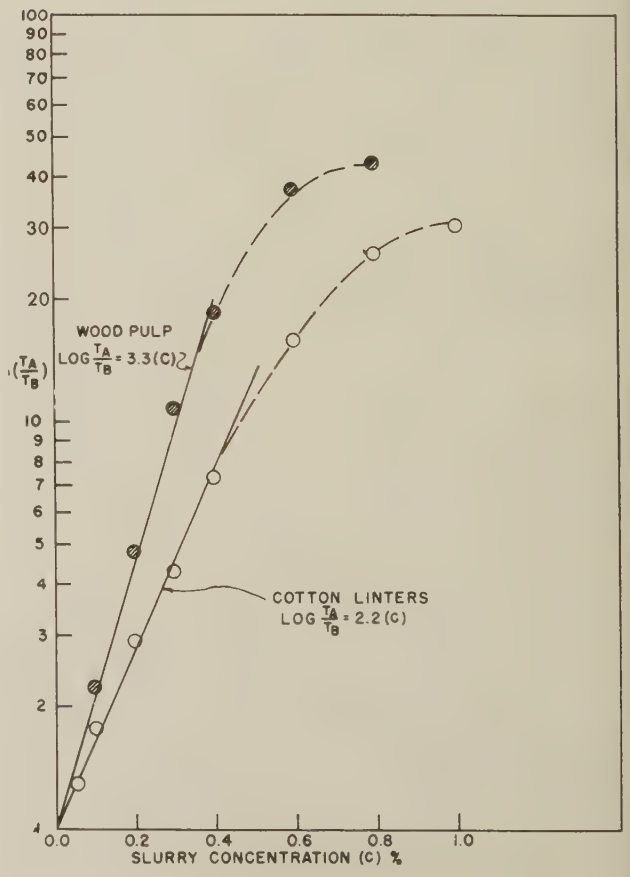

Fig. 2. Light transmission calibration

Table II. Acetylation Clearing Rate Data

2.0 g. pretreated cotton linters: 0.023 g. unactivated cotton linters

Time, min.	T_A	T_B	*Log* T_A/T_B	*Log* T_A/T_B *(corrected for blank)*
13	79.8	8.5	0.973	0.964
20	93.7	39.1	0.379	0.370
24	97.7	80.5	0.084	0.075
29	98.4	90.0	0.039	0.030
34	98.8	92.0	0.031	0.022
39	97.6	89.7	0.037	0.028
43	98.4	91.8	0.030	0.021
57	98.5	91.5	0.032	0.023
Blank	100.0	97.8	0.009	...

in which k for:

Cotton linters = 2.2
Wood pulp = 3.3

The average diameter of the cotton linters particles which formed on acid degradation was less than the average diameter of the wood pulp particles. The ranges, by microscopic examination, were from 7 to 41μ for cotton and 14 to 63 μ for wood pulp. Prolonged acid degradation did not alter these particle size distributions. This particle size range difference (7) accounts for the difference in light scattering between the two cellulose types.

Now if first order reaction kinetics are assumed, i.e., the rate of disappearance of unacetylated cellulose, c, is proportional to the amount remaining, one would expect a straight line plot of Log (c) versus t. Incorporating the Beer-Lambert relationship established previously one would expect a straight line plot of Log (T_A/T_B) versus t.

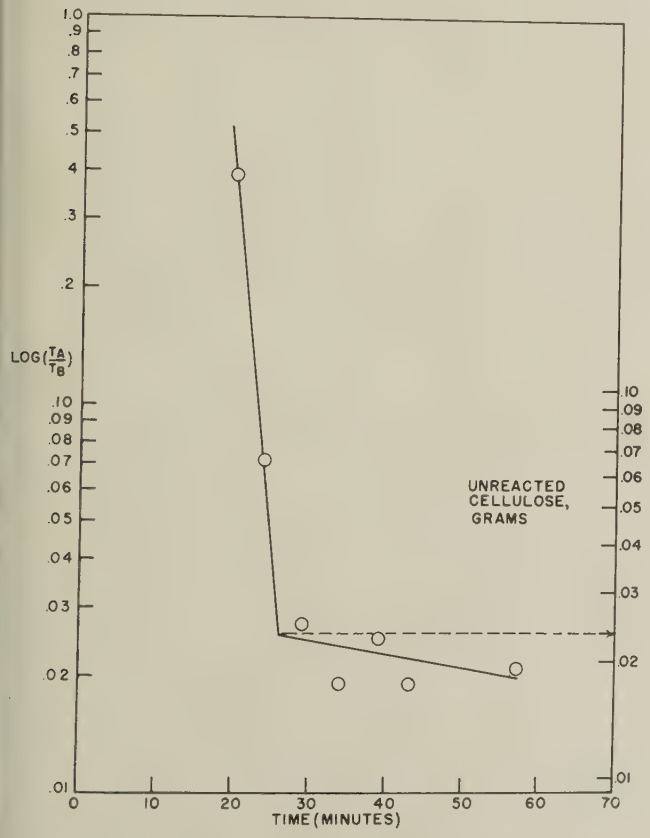

Fig. 3. Acetylation rate

2.0 g. activated linters; 0.023 g. unactivated linters.

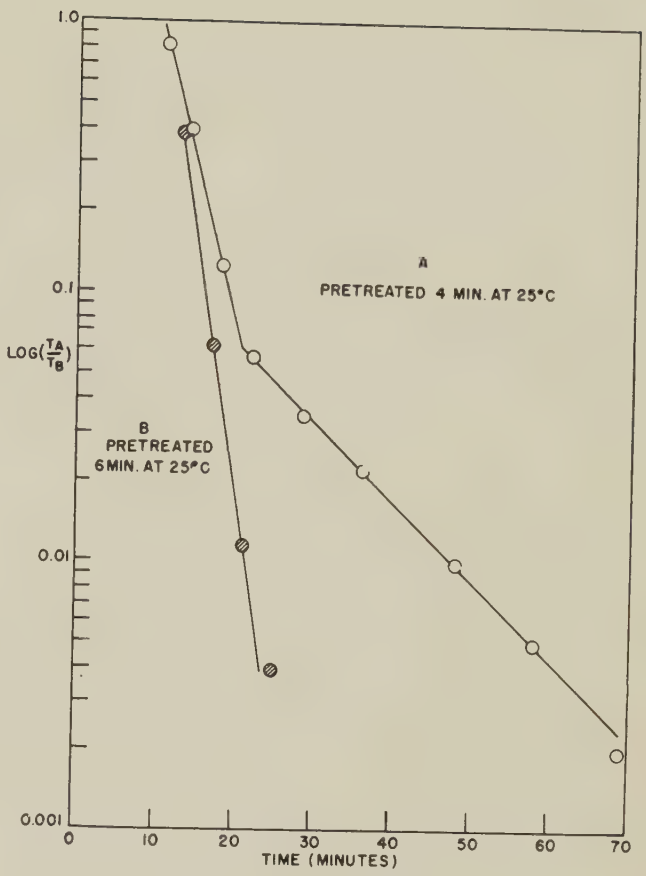

Fig. 4. Acetylation rate

A check of the calibration procedure was made by conducting an acetylation in which 0.0234 g. of unpretreated cotton linters was mixed with 2.0 g. of pretreated cotton linters at the start of the acetylation reaction. The total volume of the reaction mixture was 200 ml. The optical transmission data are shown in Table II. By plotting Log (T_A/T_B) versus time on semi-log coordinate paper (Fig. 3) a sharp break is observed at Log (T_A/T_B) = 0.0255 which is equivalent to 0.023 g. of cellulose in suspension.

The usefulness of this procedure can be illustrated by the data in Table III and Fig. 4. In Fig. 4, line A represents the light transmission data for the run as specified in Table III. Note the break corresponding to 0.6% of the cellulose incompletely activated. The same pulp pretreated for 6 min. at 25°C. gave line B, showing complete activation.

In the subsequent work, in which various celluloses were to be compared, the swelling stage was standardized at 15 min., adequate for all the cellulose samples. So long as the pretreatment was adequate for complete

Table III. Proportions for High Dilution Acetylation Procedure

Swelling stage
  4.0 g. wood pulp (8% H_2O)
  1.4 ml. acetic acid
  Agitate 4 min. at 25°C.
Catalyst sorption stage
  0.30 ml. sulphuric acid
  2.8 ml. acetic acid
  Agitate 5 min. at 25°C.
Acetylation
  150 ml. of 7% acetic anhydride 45°C.

Table IV. Cellulose Activation Procedures

	One stage	*Two stage*
Swelling stage	4.0 g. cellulose 1.4 ml. acetic acid 15 min. at 25°C.	4.0 g. cellulose 1.4 ml. acetic acid 15 min. at 25°C.
Catalyst sorption stage	None	0.30 ml. sulfuric acid 2.8 ml. acetic acid 5 min. at 25°C.

activation, i.e., the optical data showed no break, a cellulose reaction rate could be specified by a time to reach a predetermined residue. Clearing time was specified as the time required for 99.9% of the cellulose to dissolve. In the case of curve B this would be 21 min.

RESULTS

Two Cellulose Acetylation Mechanisms

Two distinct acetylation mechanisms can be observed depending on a relatively minor modification of the pretreating procedure as shown in Table IV. In the absence of acetic anhydride sulphuric acid is reversibly adsorbed from acetic acid onto cellulose (*8*). In the catalyst sorption step, Fig. 5, it distributes itself on sites throughout the cellulose fiber. In the presence of acetic anhydride sulfuric acid is chemically bound to the cellulose (*9*) at such a rapid rate that it cannot migrate into the fiber and is not present therein unless it was there beforehand. Thus in the one stage pretreatment routine, all the sulfuric acid catalyst is bound to the surface of the fiber, and the reaction proceeds from the surface only. In the two-stage pretreatment the catalyst sorbed throughout the fiber permits a more rapid overall reaction.

Acetylation of Wood Pulp/Cotton Linters Blends

In Table V acetylation clearing rate data are shown for a cotton linters sample, an acetylation grade wood pulp sample and 50/50 blends of the two. When one-stage pretreatment was used the acetylation clearing

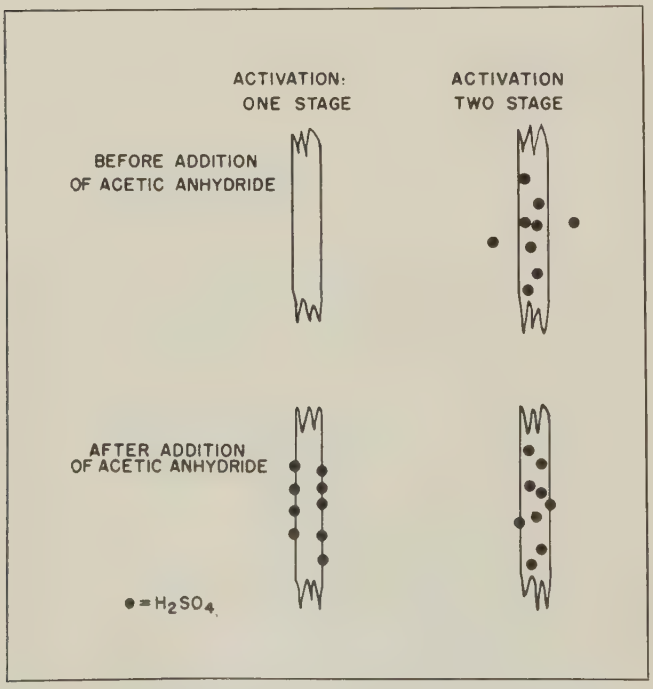

Fig. 5. Two acetylation mechanisms

Table V. Acetylation of Cellulose Blends

Composition of cellulose blend	*Time for 99.9% reaction, min.* — *One stage activation*	*Two stage activation*
100% cotton	49, 48[a]	25, 27
50/50	42, 43	31, 36
100% wood pulp	33, 33	23, 22

[a] The two numbers are the reaction times for replicate acetylations

time for the 50/50 blend fell between the 100% wood pulp and the 100% linters clearing times. When two-stage pretreatment was used, i.e., swelling with acetic acid followed by equilibrium sorption of sulfuric acid, acetylation clearing times for the 50/50 blend were significantly longer than the clearing times for the 100% cellulose type acetylations. The same results are shown in Fig. 6 in which the dotted lines indicate 2 sigma control limits for chance variation.

Figure 7 shows acetylation clearing times after two-stage pretreatment for a range of wood pulp/cotton linters proportions. Note in these data that the addition of only 10% cotton linters to wood pulp caused the acetylation clearing time to be prolonged significantly whereas the addition of 10% wood pulp to cotton linters did not prolong the reaction.

The anomalous clearing time increase for blends occurred only after two-stage pretreatment. Therefore, the probable source of the slowed reaction is an unequal distribution of the sulfuric acid catalyst between the wood pulp and the cotton linters during the catalyst sorption stage. The slowness of the clearing time of the cellulose blend containing only 10% cotton linters suggests that it is the cotton linters which does not sorb its proportionate share of the sulfuric acid catalyst.

Richter and co-workers (*8*) have reported Langmuir adsorption isotherm data for systems of cellulose, sulfuric acid and acetic acid. They report a maximum

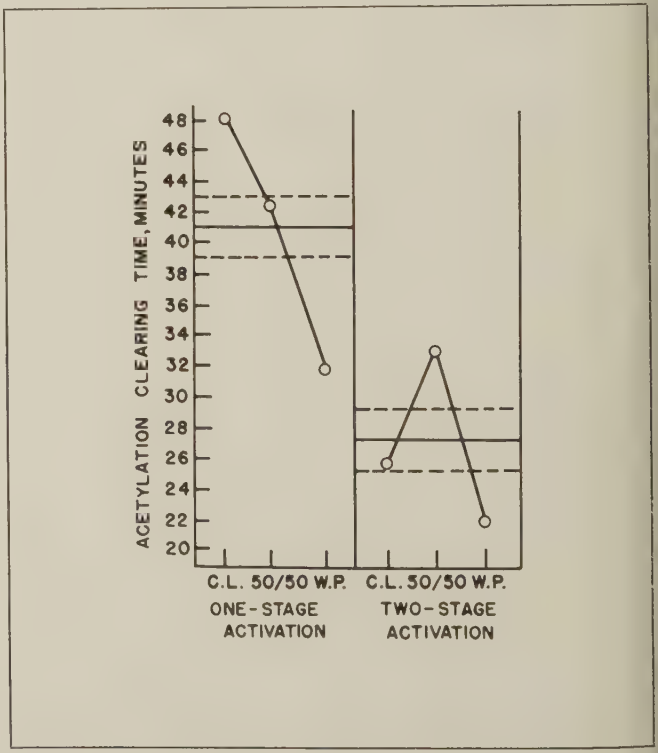

Fig. 6. Acetylation rates of blends of wood pulp and cotton linters

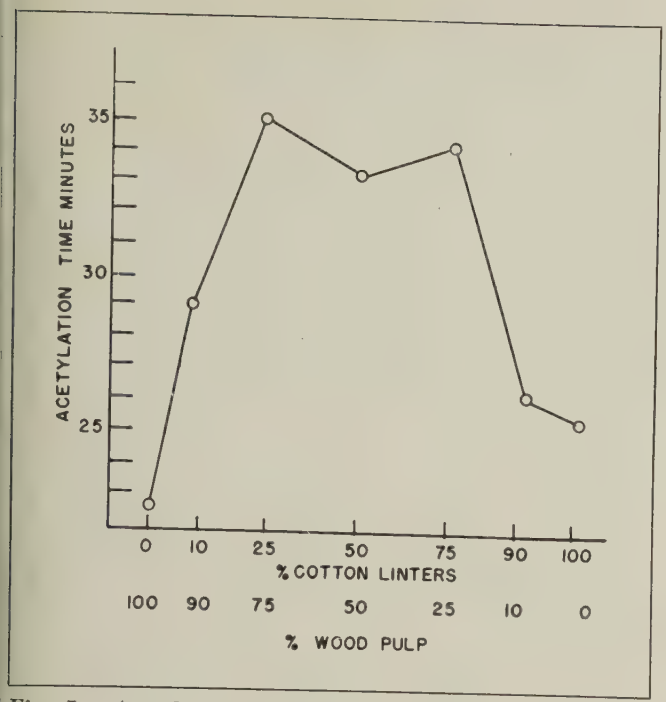

Fig. 7. Acetylation clearing times for cellulose blends

sorption capacity of 11.3% for an acetate grade wood pulp and 9.2% for an acetate grade linters, thus indicating that a disproportionate distribution of sulfuric acid in a cellulose blend is quite likely.

The critical experiment was to pretreat cotton linters and wood pulp individually using the two-stage pretreatment procedure, and then to combine them for acetylation. The results in Fig. 8 show that in this case the blend acetylation clearing time falls between the 100% pulp and 100% linters acetylation times. These results are considered as confirmation of the hypothesis that the cause of the blend clearing time anomally is the preferential sorption of sulfuric acid from one cellulose to the other.

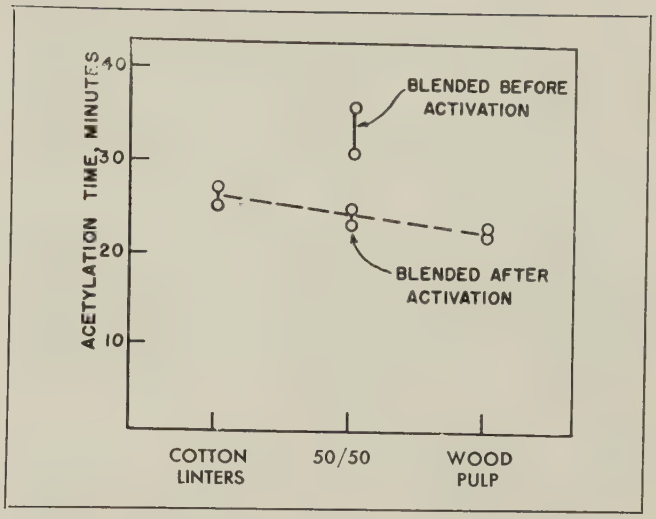

Fig. 8. Acetylation clearing times for cellulose blends

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Acknowledgment is due to Robert H. Wells who participated in the project and measured the degraded particle size ranges.

Methods for the Analysis of the Physical Structure of Clay-Starch Coating Films

DAVID J. KRASKE

This paper reports adaptations of established methods and the development of new techniques for the characterization of the physical structure of clay-starch coating films. The methods used are applicable to other coating systems. Methods used for the structural characterization of isolated coating films include low temperature gas adsorption for the measurement of surface area, pore distribution and internal void volume, and an x-ray diffraction technique for the measurement of clay particle orientation. With certain selected experiments it was shown that these methods detect and describe differences in the physical structure of coatings. The system developed is considered to be most useful in the comparison of effects of coating variables on structure. Further work with the system described herein should lead to a more fundamental understanding of variations in the end-use properties of coated papers.

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The literature of the coating field contains literally hundreds of articles relating the end-use properties of coated papers such as gloss, smoothness, and ink receptivity to the coating component and process variables. This work has been useful in the development and improvement of coated papers and will continue to serve a useful function in the field of coating technology. However, information obtained from such tests is necessarily restricted in its interpretation.

The chief difficulty with end-use testing lies in the fact that the test results, as presently obtained, have no clear-cut quantitative significance. For example, the several ink receptivity tests may be considered to be dependent upon the size of the pores in the coating, the smoothness of the coating surface, the contact angle between the coating surface and the ink vehicle,

and the pressure applied during the spreading of the ink. The results obtained from such a test may reflect changes in any one of these variables without indicating which one is responsible. Additional tests such as gloss or smoothness may shed light upon the observed variation but they cannot always provide the full answer.

In recent years, a more fundamental approach to the problems existing in the coating field has been adopted particularly with respect to dispersant performance and coating color rheology problems. The work reported in this paper provides a basis for extending this fundamental approach to an evaluation of the physical structure of clay-starch coatings. Since the structural criteria selected for use in this study have direct relationships to many of the common end-use properties, extension of this work should make possible a more thorough understanding of the uses and limitations of the end-use tests presently employed. It may also lead to a more fundamental understanding of certain phenomena occurring in the coating process: vehicle and adhesive migration, the loss of strength, and the development of gloss in supercalendering, and so on.

THE ANALYSIS OF COATING STRUCTURE

The complexities involved in the analysis of coating structure are indicated by the few attempts made in the past to characterize structure. Carson (*1*) used an air permeability technique to arrive at the conclusion that the pores in a single- and double-coated book paper average 0.30 and 0.20 μ in radius, respectively. The method used was rather sensitive to the presence of very large pores and pinholes in the sheet. Tollenaar (*2*) has developed a fluid permeability technique for pore analysis that does not differentiate between pore size and number. Other than these two investigations, nothing in the literature deals directly with the measurement of the structural characteristics of coatings.

The system for the evaluation of coating structure and the effects of certain variables on structure which is described in the remainder of this paper provides a more direct attack upon the problem of coating structure analysis than any previously reported work.

The following structural criteria were chosen for use in this study:

1. Free, or unbonded, surface area.
2. Pore size distribution.
3. Internal void volume.
4. Clay particle orientation.

Each of these physical measurements, in addition to fixing a structural feature of coatings, has a direct bearing on several end-use properties.

Surface Area

The surface area of the coatings was determined by means of low temperature nitrogen adsorption using the Brunauer, Emmett and Teller (*3*) method of data interpretation. The apparatus and techniques used were identical to those reported by Haselton (*4*) and Merchant (*5*). The size of the isolated coating samples used in these experiments was necessarily small and made requisite great care in the determination of the nitrogen adsorption isotherm. The method has excellent reproducibility but the area values obtained must be qualified as "area accessible to nitrogen molecules."

Pore Distribution and Void Volume

The pore distribution and the internal void volume of coatings may be obtained by several methods; electron microscopy (*6*), mercury intrusion (*7*), gas permeation (*8*), and gas adsorption (*9*). Gas adsorption, which has been experimentally checked against electron microscopy and mercury intrusion methods (*6*, *7*), was used in this work. The Pierce method of data interpretation (*9*) was used in spite of certain slightly objectionable details.

Again, the techniques and apparatus used were those described by Haselton (*4*) and Merchant (*5*) with only slight modifications. It was found to be necessary to start the desorption branch of the isotherm from the point of complete sample saturation in order to accurately locate the curve (*10*). The useful desorption data were obtained at values of relative pressure between 0.95 and 0.99. Care was necessary to assure that each desorption reading was taken at equilibrium. The estimated precision of the pore data obtained from the desorption isotherms varies from 6% at a pore radius of 0.1 μ to 3% at 0.03 μ radius. Above a radius of 0.10 μ the precision rapidly deteriorates.

It should be noted that the pore distributions obtained by the Pierce method are based upon effective circular radius. Actually, the pores in coatings may exist in a variety of shapes and the results presented here must be evaluated with this in mind. The gas adsorption method will detect pores of at least 0.50 μ radius but is not suited for quantitative analysis of pores of this size.

The internal void volume of isolated coatings was obtained by extrapolation of the desorption branch of the nitrogen sorption isotherm to the saturation axis. This procedure yields the volume of gas which, when condensed, will just fill the pores of the coating sample. This value can be recalculated in terms of the void volume of the coating. Ries and co-workers (*11*) have checked void volumes determined by this method against mercury displacement values and found good agreement. In the present work, a similar agreement was obtained between the nitrogen sorption and mercury methods when a heavyweight coating was examined. Attempts to check these two methods with lightweight coatings was unsatisfactory due to the fact that the surface roughness of lightweight coatings contributed substantially to their void volume as determined by mercury displacement.

Clay Particle Orientation

The analysis of clay particle orientation in coatings was based on x-ray diffraction principles. Similar methods have been extensively used in the field of metalworking. The method used in this work is an adaptation and extension of a technique originally developed by Field and Merchant (*12*).

All regularly, minutely spaced planes (or, more correctly, atoms) are capable of diffracting an x-ray beam incident upon them at certain angles. The angle at which diffraction occurs is related to the spacing of the crystal planes through Bragg's law (*13*):

$$n\lambda = 2d \sin \theta$$

where θ is the angle between the incident beam and the surface of the crystal plane, d is the distance between the crystal planes in A, λ is the wavelength in A of the x-rays employed and n is the diffraction order. The intensity of the diffracted x-ray beam at an angle θ is proportional to the number of crystal planes in the mass of material properly oriented for diffraction (see Fig. 1).

The kaolin clays used in paper coating have a crystal structure which is represented schematically in Fig. 2 (*14*). The familar platelike form of the kaolin particle (Fig. 3) is composed of many of the structural units shown in Fig. 2 arranged in such a manner that the (001) crystal plane is parallel to the large surface of the clay particle (*14*). The ratio of the greatest diameter of the clay particle to its thickness may range from 3 to 17 (*15*).

Referring again to Fig. 1, case I represents the situation normally found in x-ray diffractometers; that is, the specimen surface forms an angle θ with the incident x-ray beam. Therefore, diffraction intensity recorded with the sample in this position may be assumed to be from those particles which are oriented with their large surfaces parallel to the plane of the specimen surface. In case II the specimen has been tilted to an angle α to its normal position; diffraction now occurs from those kaolin particles oriented such that their large surfaces make an angle α to the specimen surface. It is reasonable that a definition of the extent of orientation of the clay particles, with respect to the plane of the specimen surface, may be obtained by measuring the x-ray intensity diffracted from the coating sample

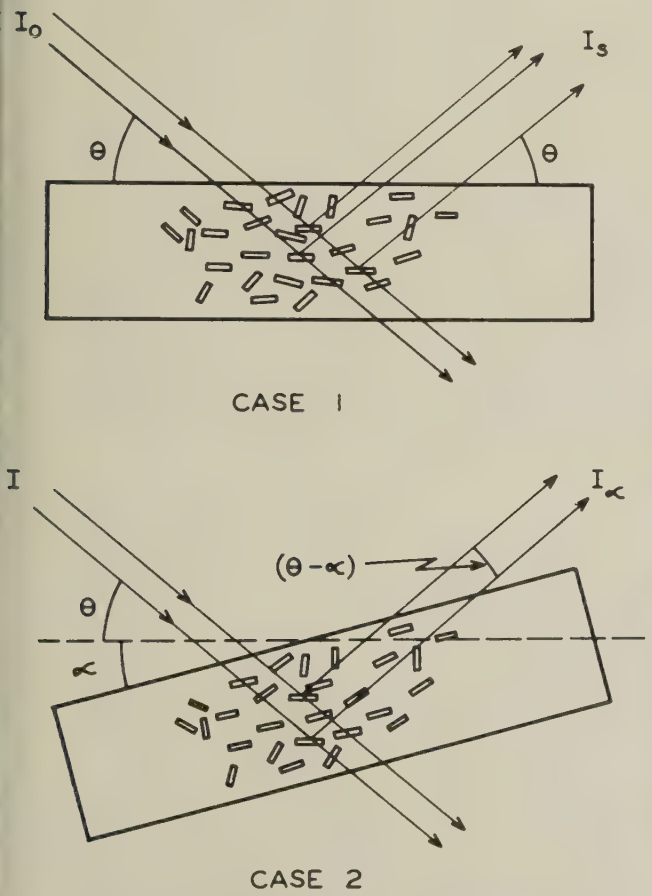

Fig. 1. Diffraction in crystalline powders

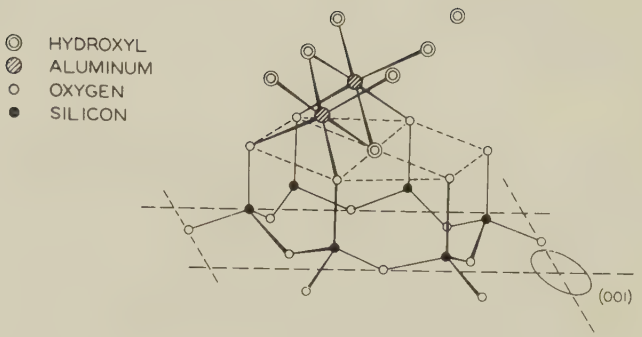

Fig. 2. Kaolinite crystal structure (taken from Brindley, 14)

when it is positioned at various values of the tilting angle.

Matter absorbs x-rays to an extent dependent upon the wavelength of the x-rays and certain characteristics of the matter. Since the path length of the x-rays in the sample varies with the tilting angle α, it is necessary to correct for differences in the absorption of x-rays when the sample is tilted.

Let

I_0 = the incident x-ray intensity
I_a = the incident x-ray intensity at point A
I_s = the diffracted intensity in the normal case
I_α = the diffracted intensity in the general case
dx = a small increment of sample thickness along path x
D = the fraction of the incident x-rays scattered per unit thickness of the sample
x = the path length of the incident x-rays in the sample
y = the path length of the diffracted x-rays in the sample
t = the thickness of the sample in centimeters
e = the base of natural logarithms
μ = the linear absorption coefficient of the material in the specimen
θ = a convenient Bragg angle, in degrees
α = the tilting angle in degrees

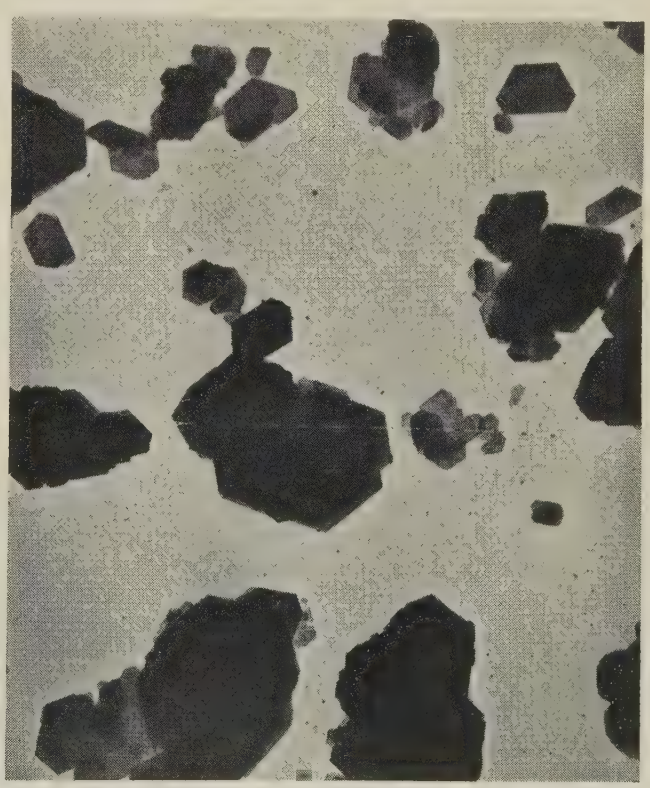

Fig. 3. Electromicrograph of kaolin clay 1.0 to 2.0 mμ fraction. 8000×

Working with the general situation (Case II, Fig. 4) the x-ray intensity reaching point A is

$$I_a = I_0 e^{-\mu x} \qquad (1)$$

The incident beam, in passing through the small increment of thickness dx at point A, contributes a minute fraction D of its energy to the diffracted beam

$$\text{at point } A \qquad dI_\alpha = I_a D dx = I_0 e^{-\mu x} D dx \qquad (2)$$

The portion of the beam which is diffracted at point A loses some of its energy in traversing the path y from point A to the surface of the sample

$$\text{at the surface} \qquad dI_\alpha = (I_0 e^{-\mu x} D dx) e^{-\mu y} \qquad (3)$$

This equation may be simplified by recognizing that x and y are related through θ and α.

$$y = x \sin(\theta + \alpha)/\sin(\theta - \alpha)$$

so that $y = xM$, where $M = \sin(\theta + \alpha)/\sin(\theta - \alpha)$. Then

$$\text{at the surface} \qquad dI_\alpha = I_0 D e^{-\mu x} e^{-\mu x M} dx \qquad (4)$$

Now, if it is assumed that dx is infinitesimally small, it is possible to collect terms and integrate equation (4), for $x = 0$ to $x = \infty$.

The development and derivation to this point is that presented by Field and Merchant (*12*). These

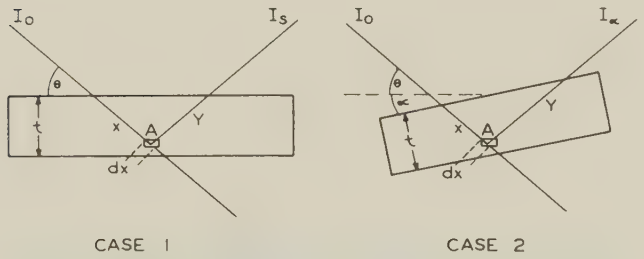

Fig. 4. X-ray paths in thin specimens

investigators were interested only in specimens which were infinitely thick to x-rays. Continuation of their derivation leads to the following equation which is useful in obtaining correction factors for x-ray absorption in samples which are infinitely thick to x-rays and sample is tilted by angle α.

$$\frac{I_\alpha}{I_s} = \frac{2}{1 + M} \qquad (5)$$

In the case of typical clay-starch coatings it can be shown that a coat weight of approximately 300 lb. per ream (25 × 38 × 500) is required to fulfill the conditions of the Field and Merchant method when Cu-Kα x-rays and the Bragg angle $\theta = 18.8°$ are used. Since this condition is not in the usual range for coated papers, it was necessary to modify the correction equation presented by these authors in the following way to be useful for lighter or thinner coating weights.

The terms in equation (4) are collected and the indicated integration is performed from $x = 0$ to $x = x$ rather than from $x = 0$ to $x = \infty$.

$$I_\alpha = I_0 D \int_0^x e^{-(1+M)\mu x} dx \qquad (6)$$

and

$$I_\alpha = \frac{I_0 D}{\mu[1 + M]} (1 - e^{-(1+M)\mu x}) \qquad (6a)$$

From consideration of Fig. 4, it is evident that in the case of a thin specimen both x and y change as α is varied. It is well therefore to express x as a function of the thickness of the specimen t.

$$x = t/\sin(\theta + \alpha)$$

Then,

$$I_\alpha = \frac{I_0 D}{\mu(1 + M)} (1 - e^{-(1+M)\mu t/\sin(\theta+\alpha)}) \qquad (7)$$

Now, at $\alpha = 0$ (Case I, Fig. 4) $M = 1$ and $\sin(\theta + \alpha) = \sin\theta$,

$$I_{\alpha=0} = I_s = \frac{I_0 D}{2\mu} (1 - e^{-2\mu t/\sin\theta}) \qquad (8)$$

From equations (7) and (8) it is possible to form the following ratio

$$\frac{I_\alpha}{I_s} = \frac{2}{(1 + M)} \frac{(1 - e^{-(1+M)\mu t/\sin(\theta+\alpha)})}{(1 - e^{-2\mu t/\sin\theta})} \qquad (9)$$

Equation (9) represents the ratio of intensities which would be expected at any value of α compared to the measured intensity at $\alpha = 0$ for a perfectly randomly oriented crystal powder or suspension. That is, the ratio of measured intensities that may be expected due to absorption differences arising from tilting of the thin specimen.

In order to correct measured intensities it is simply necessary to divide them by the value of the ratio calculated from equation (9). The quotient obtained is then directly comparable to the intensity measured at $\alpha = 0$ or to any other similarly corrected intensity measured at a finite value of α.

It should be noted that equation (9) reduces to the Field and Merchant expression as the value of the product μt becomes large.

The experimental proof of the validity of equation (9) was obtained quite simply. Decker, Asp and Harker (*16*) have shown that the silver halide particles in exposed x-ray film are randomly oriented. In the present work the emulsion containing the silver halide particles was stripped from two exposed photographic films by soaking the films in chloroform. The emulsion specimens were then mounted in a Norelco x-ray diffractometer and examined over a range of tilting angles. The measured intensity values were corrected for ab-

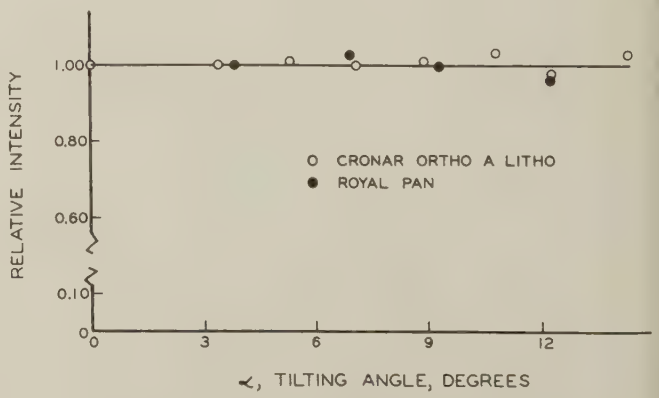

Fig. 5. Randon crystal orientation profile, photographic emulsions

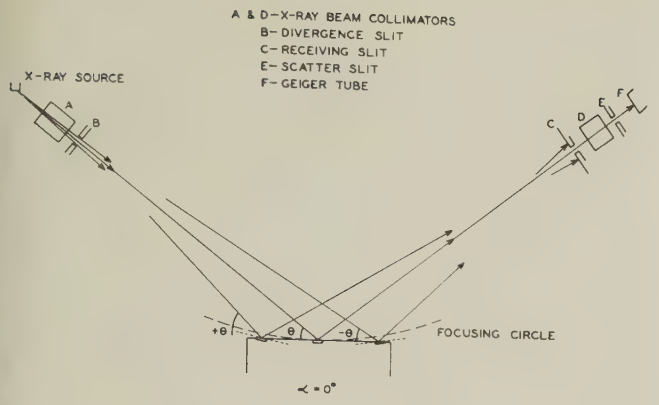

Fig. 6a. Diffractometer geometry, normal position

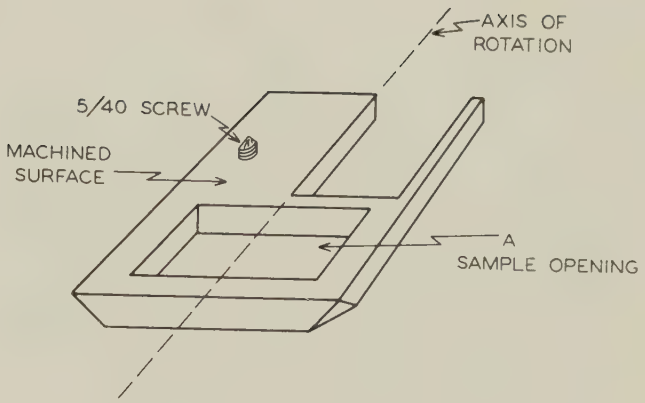

Fig. 7. Specimen holder

sorption losses according to equation (9). The value of the product μt was measured by inserting the emulsions in low intensity x-ray beams and measuring the decrease in x-ray intensity. The value of the product μt was then calculated from the following equation

$$I = I_0 e^{-\mu t}$$

where the symbols have the same meaning as indicated previously.

The results of this experiment with film emulsions are presented in Fig. 5 where the ratio of the corrected intensity measured at $\alpha = \alpha$ to the intensity measured at $\alpha = 0$ (this ratio is hereafter referred to as the *relative intensity*) is plotted against the tilting angle. The straight line of zero slope obtained by this procedure is characteristic of a randomly oriented crystal powder or suspension. This experiment may be regarded as sufficient proof of the validity of equation (9).

Figure 6a shows the essential features of the geometry of the Norelco x-ray diffractometer with the specimen positioned at $\alpha = 0$. Because the sample surface is flat there is some defocusing of the diffracted x-ray beam. Some of the beam does not reach the Geiger tube counting device due to the presence of the receiving and scattering slits. In Fig. 6b the specimen has been positioned at $\alpha = 15°$. Comparison of the diffracted beam traces in these two illustrations shows that tilting the sample causes additional defocusing of the diffracted x-ray beam. If the receiving slit (c) is removed and the scatter slit (e) is enlarged sufficiently, all of the diffracted radiation is received by the Geiger tube regardless of the value of the tilting angle, α.

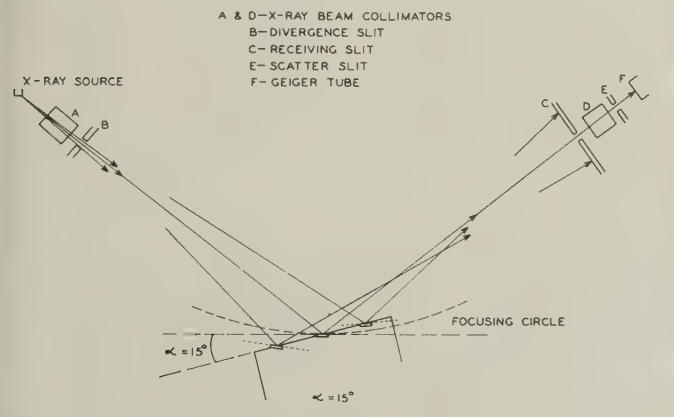

Fig. 6b. Diffractometer geometry specimen tilted 15°

However, if resolution considerations require the use of the standard slit system some distortion is introduced into the results of the orientation measurements. Within the precision of this method, if θ and the slit system are held constant through a series of measurements the error or distortion introduced is constant.

Specimens of the coating to be examined for orientation are mounted over opening A of the specimen holder shown in Fig. 7. The coating pieces are held in place with a thin layer of cement. "Composite" samples may be prepared by the addition of more coating pieces from the underside of the specimen holder. These are cut to the size of the opening and are held in place by tiny drops of cement applied to the corners of the opening. The use of "composite" samples tends to average out variations in test results due to within coating sample variation in orientation.

The specimen holder is positioned in the Norelco x-ray diffractometer as shown in Fig. 8. Care must be taken that the axis of rotation of the specimen holder coincides with the center line of the diffractometer sample clamp. The tilting angle is varied by means of the 5/4D screw shown in Fig. 7. The actual value of angle α is obtained through measurements with a cathetometer of the elevations of the corners of the specimen holder after it has been positioned in the diffractometer. The diffractometer is started and

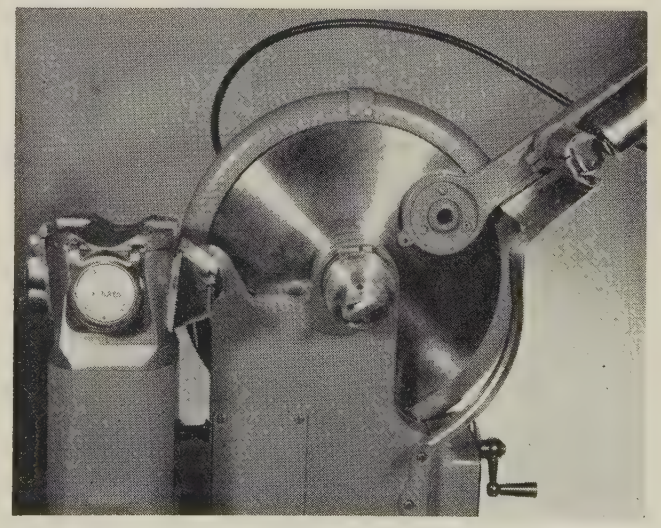

Fig. 8. Norelco x-ray diffractometer specimen positioned at $\alpha = 15°$

allowed to traverse the diffraction peak area twice. [In this work the (003) kaolin-crystal plane, θ = 18.8°, was used in the examination of all coatings.]

The x-ray intensity received by the Geiger tube is recorded on a strip chart under constant conditions. The area under each diffraction peak, after correction for background radiation, is taken as a measure of the diffracted x-ray intensity. Through the use of certain machine constants this area can be converted to counts delivered by the Geiger tube. Intensity values obtained by this method are corrected for absorption losses according to equation (9). The value of the linear absorption coefficient, μ, may be obtained for the specific material examined from tables of mass absorption coefficients of the elements (*13*). A knowledge of the chemical composition of the material is required. An alternative procedure involves the measurement of the product μt as previously described.

It is obvious that the greater the numerical value of the slope of the straight lines presented in Fig. 9, the greater the extent of the clay particle orientation in the coating sample. The values of orientation reported in this study are the slopes of plots such as those in Fig. 9. The experimental values were obtained using a standard slit system and, therefore, they contain the defocusing error previously discussed. Addition of an experimentally determined correction factor, 0.009, to the reported values converts them to an absolute basis.

The straight-line plots, shown in Fig. 9, as indicative of the results obtained with clay coatings, probably represent the steepest slope of a distribution curve. Accuracy limitations preclude work at values of α less than 1° and high x-ray absorption in the sample obscures results obtained at values of α greater than 15 to 16° so that the actual definition of such a distribution curve is impractical at the present.

The effect of machine or cross direction (with respect to the coating operation) may be evaluated by controlling the direction in which the specimen is mounted on the specimen holder. With the coatings used in this work no significant difference was found for orientations measured in the two directions.

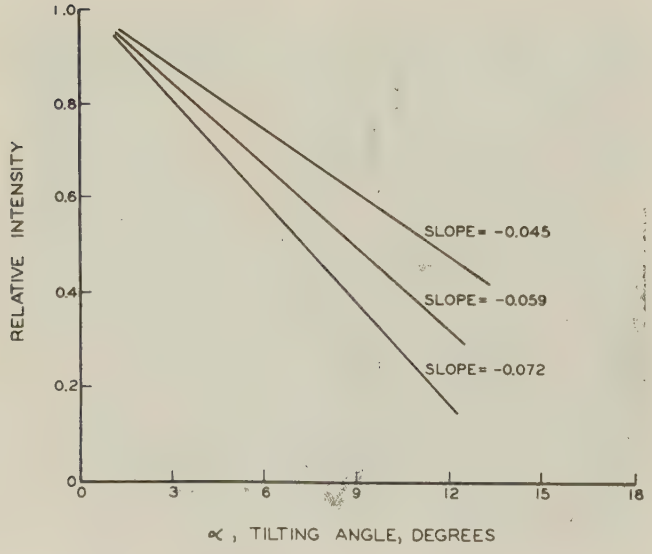

Fig. 9. Orientation profiles, clay coatings

Coating Isolation

The rawstock may have important effects upon the structure of coatings during the deposition and drying of the coating films. It serves only, however, to complicate the experimental phase of coating structure analysis. The solution to this problem is the use of a coating rawstock from which the coating may be isolated, in pieces of convenient size, without altering the coating structure. To this end, attempts were made to form handsheets from chloroform-soluble fibrous cellulose acetate. Difficulty was encountered in the sheet-forming stage and this approach was abandoned.

Millipore filter, a product of the Millipore Filter Corp., was finally selected as the rawstock to be used in this study. This material, a porous nitrocellulose sheet, is soluble in ethyl acetate. Ethyl acetate does not swell starch (*17*) and therefore treatment of coatings with this solvent should not effect coating structure. The filter material is fragile, has a very smooth surface, pores whose long axes are perpendicular to the plane of the filter and a low but finite contact angle with water. It is best considered as an idealized coating rawstock.

The coating isolation procedure consists simply of a 7-stage countercurrent washing of the coated filter in ethyl acetate. The washing was accomplished in 15-cm. culture dishes which has been filled with ethyl acetate. A 10-mesh screen was placed in the bottom of each dish to facilitate transfer of the coating pieces from one bath to the next. The coating pieces, about 1-in. square, were placed in the first bath coating side down. After 10 min. the screen was lifted from this first bath and transferred to the next. A second coating sample was then placed in the first bath. After these first two steps the baths were moved countercurrently to the coating so that clean ethyl acetate was used in the final bath of each coating batch. The Millipore filter was largely removed in the first two baths.

In the fourth or fifth bath a thin film or mat was observed to break loose from the surface of the coating which had been in the rawstock coating interface. After separation and washing, this material stained blue with iodine solution and was assumed to be that portion of the starch adhesive which had migrated into the rawstock. If portions of this mat adhered to the coating surface in the seventh bath, they were removed with a small brush.

After the coating was removed from the last bath it was allowed to air dry. Work with this method showed that coatings as light as 4 lb. per ream could be isolated in a coherent form although 8 to 10 lb. per ream appeared to be the practical limit.

The Millipore filter was essentially completely removed from the coating. Examination of the solvent used in an eighth bath by the Rayleigh Interferometer showed that no further dissolution of the rawstock had occurred in this bath. The brown ring test for nitrates was performed on a sample of isolated coating with negative results. Control tests showed that this indicated that *less* than 0.02%, by weight, of the Millipore filter remained on the coating.

Internal bonding strength tests (*18*) on coated papers which had been soaked in ethyl acetate showed that the isolation treatment occasioned no loss in coating

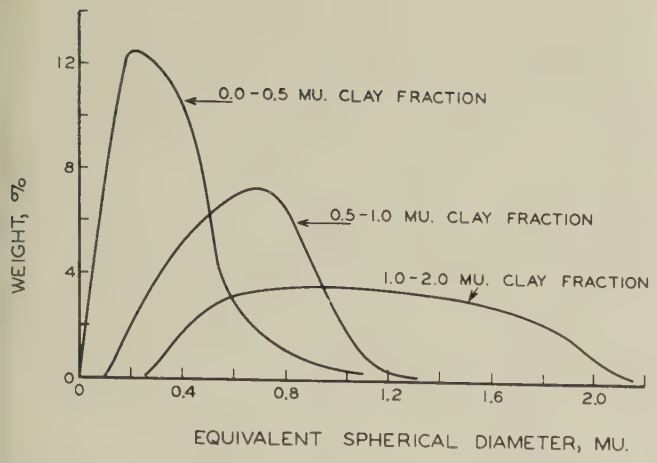

Fig. 10. Particle size distribution, clay fractions

strength. Further tests on a coating which had been mechanically isolated from aluminum foil showed that the ethyl acetate treatment had no measureable effect on the surface area, the internal void volume, the pore distribution, and the clay particle orientation of this clay-starch coating. The coating examined was rather heavy (100 lb. per ream) and was isolated from the foil by the simple expedient of twisting strips of the coated foil.

Materials and Coating Preparation

The clays used in this work were three particle size fractions of "Lustra" coating clay supplied by Southern Clays Inc. The particle size distributions of these clay fractions are presented in Fig. 10. The clay slips were dispersed with 3-milliequivalents of sodium tetraphosphate per 100 g. clay and were adjusted to a pH of 8.0 with 1.0 *N* NaOH.

The adhesive used throughout this work was an oxidized starch, Stayco M, supplied by A. E. Staley Co. The starch was cooked at 25% solids following the schedule recommended by the supplier. The starch slurry was heated to 96°C., held at this temperature for 30 min. and then cooled to 50°C. and held at this lower temperature until used.

The coating colors were prepared by adding the required amount of starch slurry to a dispersed clay slip. The color was passed through a hand-operated dispersion mill (Chicago Apparatus, no. 22950) and then was screened through a 325-mesh screen. All coating colors were prepared with a clay:starch ratio of 100:12. The solids content was held at 55% after the pH of all colors was adjusted to 8.0.

The Millipore filter was coated using Bird applicator bars. The coated sheets were allowed to air dry at 73°F. (23°C.) and 50% R.H.

INVESTIGATIONS OF COATING STRUCTURE

This portion of the study was directed primarily at demonstrating the usefulness and capabilities of the structure analysis techniques previously described. A secondary objective was that of gaining an insight into the structure of various coatings and the way in which structure is affected by certain selected independent variables. The scope of this work is far too narrow to provide a basis for general conclusions concerning coating structure.

Table I. Coating Replication

	Coating number B	Coating number J
Clay particle size, μ	0.5–1.0	0.5–1.0
Rawstock pore size, μ	0.45	0.45
Coat weight, lb./ream	21.1	21.2
B.E.T. surface area, m.²/g.	5.9	6.2
Pore distribution		
Av. radius, μ	0.054	0.057
Distribution peak, μ	0.052	0.053
Range, 90%, μ	0.034–0.098	0.036–0.093
Void volume, ml./g.	0.171	0.177
Orientation, slope, ΔR.I./deg.	−0.062	−0.062

Coating Replication

Two coatings containing the 0.5 to 1.0μ clay fraction were prepared independently. Insofar as possible all conditions were made equal. The results of the analysis of these coatings are listed in Table I.

The differential pore distributions of these two coatings are presented in Fig. 11.

It is evident from the data presented that the method of preparing coatings lacks complete reproducibility. The variations indicated in the data make it advisable that control or reference coatings be prepared with each experiment.

The Effect of Clay Particle Size on Coating Structure

Three coatings, each containing a different particle fraction but otherwise identical, were prepared, isolated and analyzed. The results of this analysis are listed in Table II.

The unbonded surface area of these coatings increases substantially as the particle size of the clay used in the coating is decreased. This follows logically from consideration of the size and number of clay particles per unit weight of coating.

One of the most interesting aspects of the pore distribution data is the range of pore sizes found in the coatings. Previous work by Carson (*1*) had indicated that the average pore radius of a single-coated book

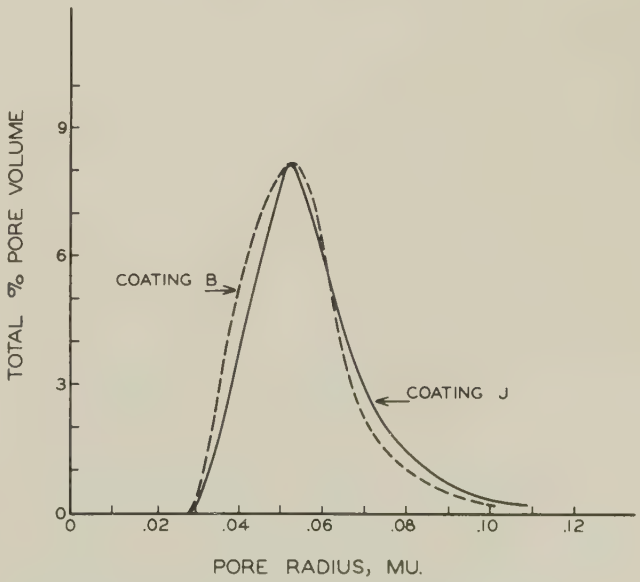

Fig. 11. Pore distributions, coatings B and J

Table II. Effect of Clay Particle Size on Coating Structure

	Coating number		
	A	*B*	*C*
Clay particle size, μ	0.0–0.5	0.5–1.0	1.0–2.0
Rawstock pore size, μ	0.45	0.45	0.45
Coat weight, lb. per ream	22.6	21.1	21.5
B.E.T. surface area, $m.^2/g.$	8.4	5.9	3.8 (6.1)[a]
Pore distribution			
Av. radius, μ	0.039	0.054	0.079
Distribution peak, μ	0.039	0.053	0.040–0.085
Range, 90%, μ	0.023–0.057	0.034–0.098	0.035–0.120
Void volume, ml./g.	0.187	0.171	0.215
Orientation, slope, ΔR.I./deg.	−0.60	−0.062	−0.045

[a] Surface area calculated from the pore distribution.

paper was 0.30μ. The present data show that the vast majority of pores in these coatings are between 0.025 and 0.10μ in radius. The sensitivity of Carson's air permeability method to very large pores and pinholes may account for this difference in pore values. Such pinholes are inherently more likely to occur when a coating is deposited upon a rough surface such as that of paper. It should be recalled that the nitrogen adsorption method can detect pores as large as 0.50μ in radius but is not suited to a quantitative evaluation of pores of this size. Pores with radii this large are, in many cases, as large as the clay particles themselves and are more aptly classified as pinholes.

The pore distributions are shown graphically in Fig. 12. It is evident that the pore distributions in coatings A and B are in a narrow range. This leads one to believe that the pore structure of these coatings is quite uniform through their thickness. In coating C, however, the pore distribution has two maxima. The surface area of this coating calculated from the pore distribution was much greater than the measured surface area. Ries and co-workers (*11*) state that this phenomena is due to the presence of constricted pores in the adsorbent; that is, pores whose body is larger than the pore opening. Although this is only fragmentary evidence, one may postulate the idealized pore structure shown in Fig. 13 for coating C. The nitrogen adsorption technique can give no clue as to whether pore opening A is located preferentially in one of the coating surfaces.

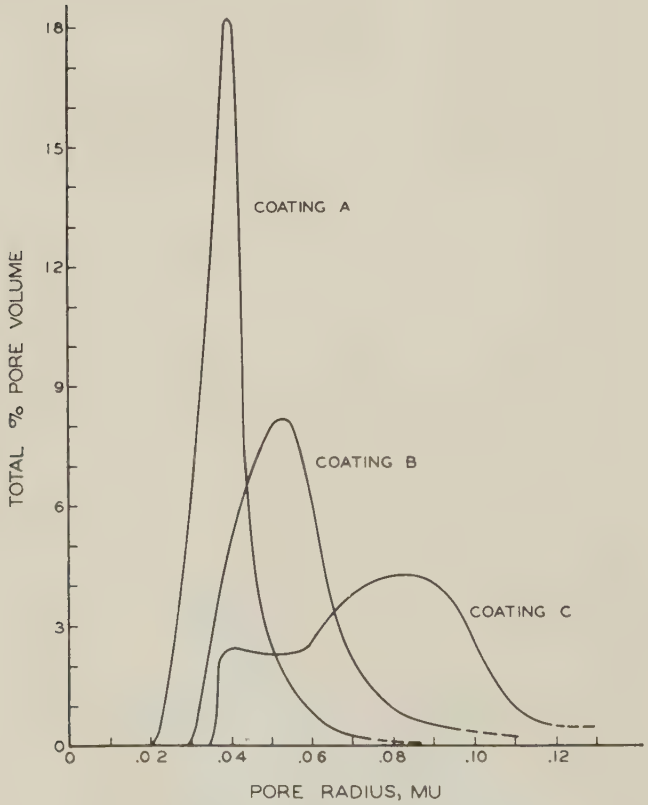

Fig. 12. Pore distributions, coatings A, B, and C

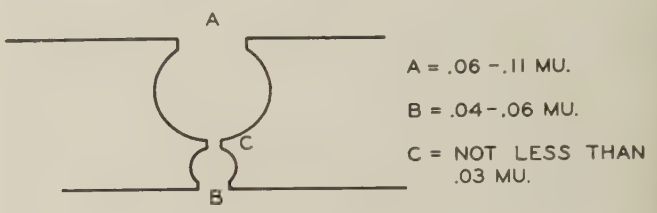

Fig. 13. Pore type, coating C

The clay particle orientation of these three coatings is of interest. The x-ray method showed that the ranking of the coatings in order of increasing orientation was C–A–B. The relatively poor orientation of the large clay particle fraction coating conceivably could result from the rapid flow of vehicle from the comparatively large interparticle spaces which should exist in this coating during the vehicle migration stage of drying. If this were the case, the particles would not have time to align themselves in the plane of the rawstock before they were "immobilized" (*19*). The comparatively large mass of these particles would offer additional resistance to alignment by fluid friction forces operative during the vehicle migration stage.

The extent of particle orientation in coatings A and B is nearly identical although the smallest clay fraction coatings consistently gave lower orientation values than the 0.5 to 1.0μ fraction. This may be due to the fact that the 0.0 to 0.5μ fraction particles are quite "chunky" when compared to the particles of the larger fraction. The length to thickness ratios of these two clay fractions, as calculated from the Muller equation (*15*) are 6.0 and 7.7 for the small and medium fractions, respectively. An additional possibility of explaining the lower degree of orientation in the 0.0 to 0.5μ fraction coating lies in the fact that many of the particles in this fraction are smaller than the pores of the rawstock upon which they are deposited. An experiment designed to check this possibility showed that tipping of clay particles into rawstock pores did not occur until the pores were almost twice as large as the greatest diameter of the clay particles.

The apparent correlation between the degree of clay particle orientation and the void volume of the three coatings listed in Table II is reasonable. This relationship was not investigated further since the reasons for its existence are obvious in basic packing theory.

The Effect of Coat Weight on Coating Structure

Three coatings, 10, 20 and 30 lb. per ream, were deposited on a Millipore filter rawstock from the same

Table III. Effect of Coat Weight on Coating Structure

	Coating number		
	D	*B*	*E*
Clay particle size, μ	0.5–1.0	0.5–1.0	0.5–1.0
Rawstock pore size, μ	0.45	0.45	0.45
Coat weight, lb./ream	10.8	21.1	33.2
B.E.T. surface area, m.²/g.	5.4	5.9	5.8
Pore distribution			
Av. radius, μ	0.063	0.053	0.056
Distribution peak, μ	0.050	0.053	0.053
Range, 90%, μ	0.037–0.105	0.034–0.098	0.035–0.093
Void volume, N_2 adsorption, ml./g.	0.171	0.171	0.176
Orientation, slope ΔR.I./deg.	−0.061	−0.062	−0.062

coating color. The coatings were isolated, analyzed, and the results of the analysis are presented in Table III.

The most interesting point illustrated by these data is the similarity between these three different coating weights. Coating D appears to have a slightly coarser pore structure than the two heavier coatings (Fig. 14) and this is reflected in the lower surface area of this coating.

Eames (*20*) has evidence that lightweight coatings lose proportionately less starch to the rawstock than do heavier coatings. It was observed that the 10.8 lb. per ream coating set, or lost its watery sheen, more quickly than the two heavier coatings. This information indicates that the slight increase in coarseness of the pore structure of the light coating may be explained in perhaps two possible ways.

(a) The vehicle drains from the lightweight coating into the rawstock more rapidly and immobilizes the clay particles before they have an opportunity to orient as well as they do in heavier coatings. (This decrease in orientation shows up in the data given for the three coatings but, statistically, the values reported are identical.) The less well oriented structure provides the base for a coarse pore system.

(b) The proportionately larger amount of adhesive retained in this coating is evidently sufficient to fill in or establish bonds across small interparticle spaces to the extent that the free surface area remaining is slightly lower than in the heavier coatings.

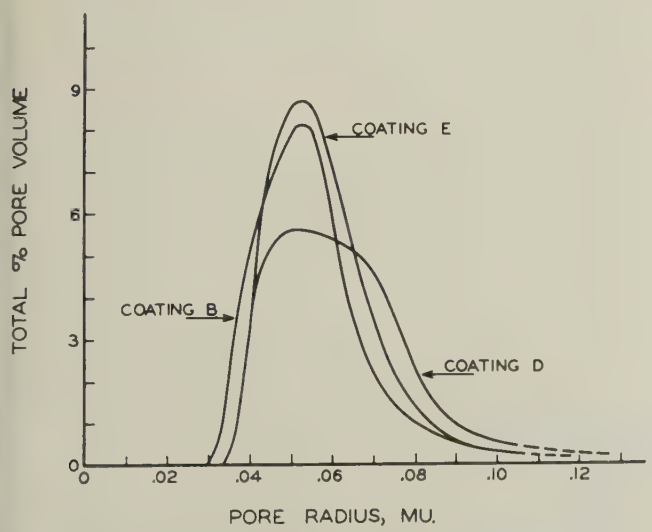

Fig. 14. Pore distribution, coatings D, B, and E

The Effect of Supercalendering on Coating Structure

Twenty-four sheets of Millipore filter were coated with one coating formulation and then were air dried under standard conditions. The coated sheets were separated into three groups of eight sheets each. One of these groups was analyzed directly. A second group was passed through one nip of a laboratory supercalender at a nip pressure of 800 lb. per linear inch. The third group was passed through 3 to 4 nips of the supercalender at the same pressure. The specular gloss of the various coated sheets was measured with the Bausch and Lomb glossmeter. The coatings were isolated and the printing opacity of the coating layer was measured with the General Electric Recording Spectrophotometer using a reduced sample aperture. The structure of the coatings was analyzed by the methods described. The results of this experiment are listed in Table IV.

Before discussing these data it should be noted that there exists a possibility that Millipore filter and paper behave entirely differently in the supercalendering process. Superficially, at least, the Millipore filter sheets responded to supercalendering as do coated papers. The caliper of the coated sheets was reduced, the coated surface was smoothed considerably and the sheets were made visually less opaque. Insofar as this preliminary work is concerned it appears reasonable to assume that the forces operating on these coatings in supercalendering were essentially the same as would affect coatings laid on paper.

Table IV. The Effect of Supercalendering on Coating Structure

	Coating number		
	J	*K*	*L*
Clay particle size, μ	0.5–1.0	0.5–1.0	0.5–1.0
Rawstock pore size, μ	0.45	0.45	0.45
Coat weight, lb./ream	21.2	20.8	20.6
Supercalendar nips	0	1	3–4
Gloss, %	58.5	70.0	72.5
Opacity, 450 mμ, %	86.1	89.5	91.5
B.E.T. surface area, m.²/g.	6.2	6.4	6.6
Pore distribution			
Av. radius, μ	0.057	...	0.059
Distribution peak, μ	0.052	...	0.059
Range, 90%, μ	0.032–0.093	...	0.027–0.081
Void volume, N_2 adsorption, ml./g.	0.177	...	0.177
Orientation, slope ΔR.I./deg.	−0.062	−0.065	−0.069

A continuous increase in the degree of pigment particle orientation was evident as the amount of supercalendering was increased. This increase in particle orientation would, perhaps, have been more pronounced if the surface of the Millipore filter rawstock had been roughened before it was coated.

The realignment of clay particles was accompanied by an increase in the free surface area of the coatings. This was to be expected since it is unlikely that significant realignment of clay particles could occur without the rupture of pigment-adhesive and/or adhesive-adhesive bonds.

More interesting is the change in pore distribution occasioned by supercalendering. From Fig. 15 it is apparent that supercalendering causes both a compression of some pores and an enlargement of others. Both could occur as the result of the mechanical rup-

ture of bonds. It is equally interesting that this shift in pore distribution occurred without a simultaneous change in the void volume of the coating. This indicates that these coatings must possess certain properties of an elastic solid since any compression of the coating must have been reversible in spite of the fact that the distortion of the coating was sufficient to cause rupture of bonds.

It has been a common observation that coated papers suffer a loss in opacity during the supercalendering operation. The information presented here indicates that the loss in opacity probably occurs solely in the rawstock since the opacity of the isolated coating was actually increased by supercalendering. This concurs with Tollenaar's finding (*2*) that supercalendering of coated papers produced a compression of some inner layer of the sheet.

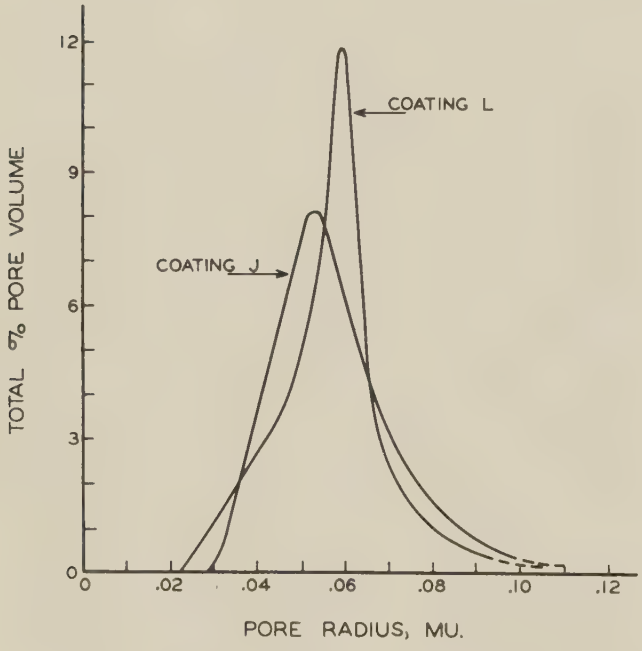

Fig. 15. Pore distribution, coatings J and L

SUMMARY

During the course of this work several methods were developed for use in the analysis of the physical structure of clay-starch coatings.

1. Low temperature nitrogen adsorption measurements were shown to be applicable in the description of the surface area, the pore distribution, and the void volume of coatings.

2. An established x-ray diffraction technique was modified to enable its use in the measurement of the extent of clay particle orientation in coatings.

3. A method was developed for the isolation of coatings in a coherent and unaltered form from an "ideal" rawstock. It was shown that the isolation treatment did not affect the physical characteristics of clay-starch coatings.

The analysis of certain coatings by these techniques showed that the methods described herein are capable of detecting and describing differences in the physical structure of coatings. Although the scope of this work was too narrow to permit broad conclusions based on these experiments, several interesting observations were recorded.

1. The pore size range of the coatings examined in this work was quite small, ranging from 0.023 to 0.105 μ in radius. These values are considerably less than those reported in the literature.

2. The pore distribution of certain coatings appeared to be strongly dependent upon the particle size of the clay used in the coating. Coating weight had but small effects upon the pore distribution of coatings.

3. The degree of particle orientation in coatings was found to be dependent upon the particle size distribution of the clays used in the coatings.

4. Supercalendering of coated Millipore filter sheets produced a realignment of clay particles, some rupture of bonds, and an interesting shift in the pore distribution of a coating. The internal void volume of the coating was not altered by supercalendering.

The greatest value of the work presented in this paper lies in the use that can be made in the future of these methods for the analysis of coating structure. The development of the relationships between the characteristics measured by these methods and common end-use properties of coated sheets should lead to a more fundamental understanding of the appearance and performance properties of coated sheets which are of interest to both the producer and the consumer.

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A Device to Count Dirt in Paper

LOWELL W. ZABEL and WILLIAM H. CUFFEY

Considerable care is taken in the papermaking process to keep the paper clean and free from specks of dirt. Any dark blemish on white paper readily attracts the eye. In the past, a visual dirt count has been based primarily on the area of the specks. To overcome some of the obvious disadvantages in such a visual system, a dirt counter has been developed to replace the subjective grading. A sample is rotated and traversed beneath a photoelectric viewing assembly. The passage of a dirt speck under this assembly causes an electrical pulse whose amplitude depends on a combined effect of area and reflectance. The system amplifies these pulses, sorts them into three groups according to pulse height, and finally displays the number of specks counted. The dirt counter is being used as a routine method on bookpaper, writing paper, and newsprint. Other experimental applications are described.

CONSIDERABLE care is taken in the entire papermaking process to keep the paper clean and free from specks of dirt. Any dark blemish on white paper readily attracts the eye even when the specks are relatively sparse. In the past, the paper has been evaluated for dirt by visually counting the specks as compared to the J. D'Arcy Clark Dirt Estimation Chart. Such a system requires specially trained operators, is fairly time consuming, and is difficult to keep in control. In order to overcome some of these difficulties, a dirt counter has been developed.

The dirt counter works in the following way: The sample is rotated and traversed beneath a photoelectric viewing assembly. The passage of a dirt speck under this assembly alters the amount of light reflected from the surface of the paper in the viewed area. This change in light is transmitted through a self-illuminating microscope to a phototube. The phototube produces an electrical pulse whose amplitude depends on the amount of light change due to the speck of dirt viewed. In general, the electrical pulse amplitude will depend on both the area of the dirt speck and also on its reflectance. In this respect the dirt counter is somewhat different from the visual dirt count method. There the primary classification was based on the area of the dirt speck. The dirt counter sorts out the resulting pulses as to amplitude and then displays the number of specks counted in each of three speck sizes over some given area of the sample. A block diagram of the system is shown in Fig. 1. A photograph of the dirt counter is shown in Fig. 2.

THE DIRT COUNTER

Sample Holder

In Fig. 3 the rudiments of the dirt counter sample holder are shown. The sample holder consists of a circular porous bronze plate which is rotated at 5 revolutions per sec. This plate is mounted on a rotating shaft of a synchronous motor. The paper sample to be tested is held against the porous plate due to a vacuum chamber beneath the porous plate. A vacuum line runs into this chamber through a rotating seal. It is, of course, desirable not to have to hold the sample flat with a piece of glass since the glass itself can become dirty or scratched and thus cause extraneous counts. The synchronous motor that rotates the sample holder is mounted on a long arm which is free to oscillate in the horizontal plane about a vertical axis through one end of the arm. The other end of the arm rides on two wheels that roll on a smooth machined surface in the horizontal plane. This arm oscillation provides the scanning of the sample in the radial direction. The radial oscillation is provided by a cam on a 17 r.p.m. motor pushing on the arm carrying the sample holder. The cam is cut so that for one revolution of the paper sample, the paper is moved radially just the width of

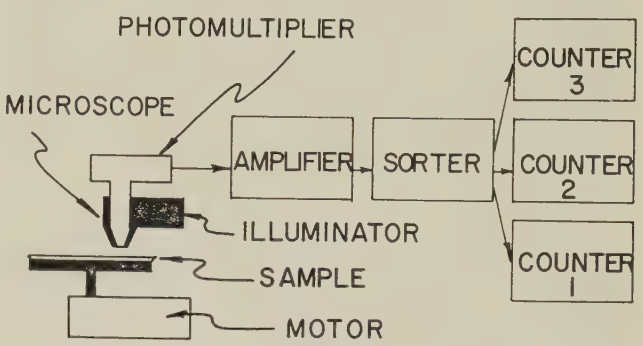

Fig. 1

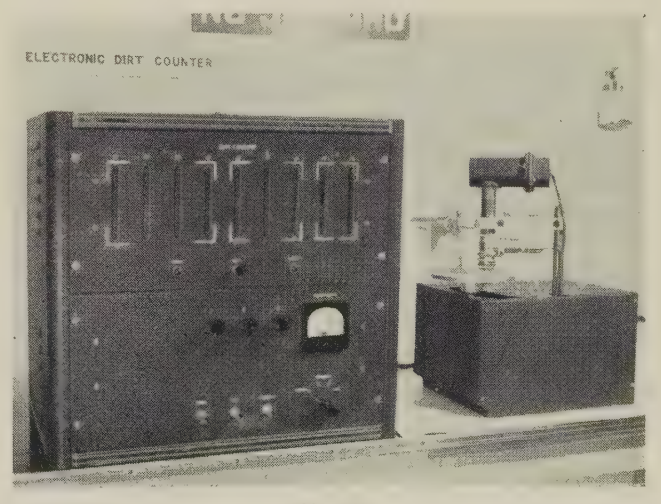

Fig. 2

LOWELL W. ZABEL, Chief, Instrumentation Laboratory, and WILLIAM H. CUFFEY, Physicist, Pioneering Research Laboratory, Research and Development, Kimberly-Clark Corp., Neenah, Wis.

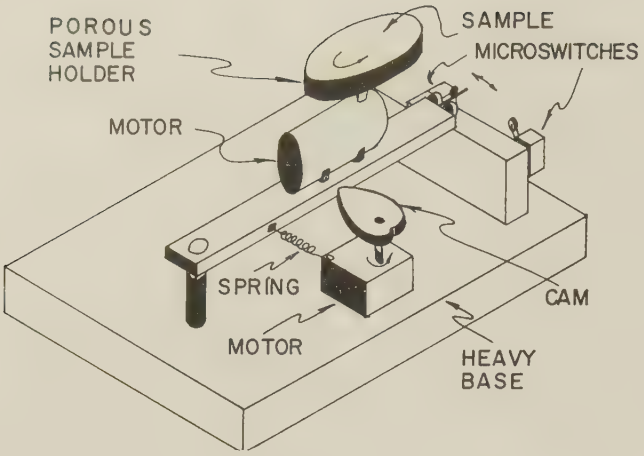

Fig. 3

the viewed area on the sheet. In this manner, the viewed area moves in a spiral path on the sample being scanned. Using this type of scanning there is no joint or seam to be blanked as one would find on a rotating drum, for example. Two microswitches, activated by the arm moving in the horizontal plane, start and stop the pulse counters. Thus, the area viewed is spatially selected. One pass at the sample looks at about 23.5 sq. in.

The Optical System

A self-illuminating microscope is used to illuminate the sample and also to form an enlarged image of the sample on a diaphragm mounted in front of the phototube. The magnification provided by the assembly is 8×. The diaphragm has a circular aperture whose diameter is 0.228 in. Thus, the area viewed on the sheet is about 0.028 in. diam. When one looks at a dirt speck by eye, one can look at the entire dirt speck. The dirt counter, however, is not so privileged for it must view the spot in the manner it passes through the viewed area. Thus, any speck of greater dimension than the viewed area or a speck that does not strike the viewed area squarely is cut up and viewed in parts. This causes the system to count too high a number of dirt specks in the lower sizes. The viewed area was chosen because of the necessity to get a favorable signal to noise ratio in the electrical system.

The Electrical System

The light reflected from the paper surface is transmitted and focussed by the microscope. That portion of the reflected light passing through the diaphragm strikes the sensitive cathode of a type 931-A photomultiplier. The passage of a dirt speck under the microscope results in a momentary reduction in the illumination of the phototube and a corresponding reduction in photocurrent. The size of this pulse depends, among other things, on the difference between the mean reflection from the paper surface and that from the dirt speck. In order to compensate for differences in average reflection from sample to sample, the amplification of the phototube is automatically varied. It is so controlled as to be inversely proportional to the mean sample reflectance. This is accomplished by varying the power supply voltage to the phototube dynodes so that the mean photocurrent is a constant. The electrical pulses formed at the photocathode are thus amplified to a greater or lesser extent depending on the average light falling on the cathode.

The pulses from the phototube are amplified by two stages of amplification and then applied to the control grid of a monostable multivibrator. This device has the property of changing conducting states any time that the control grid exceeds a preset voltage. The use of three multivibrators with parallel inputs and three independently set bias voltages permits sorting the pulses into three classes. Each multivibrator will accept all pulses greater than its preset bias. The output of the multivibrators is connected directly to three separate sets of electronic decade counters. With this system, the no. 1 channel indicates the total number of dirt specks above the threshold. The number of no. 1 specks can be determined by subtracting the no. 2 channel counts from those registered on the no. 1 channel. In a similar manner, the no. 2 dirt specks can be obtained by subtracting the no. 3 count from the no. 2 count. The no. 3 dirt specks are considered to be all dirts larger than the no. 3 channel level setting.

As with any electrical system, various unwanted signals are generated within the system. Most of these can be minimized by careful electrical and mechanical design. The most troublesome of these is the 120 c.p.s. variation in the light output of an incandescent lamp operated on 60-c.p.s. current. This signal is minimized by operating the illuminating lamp on full-wave rectified current and by setting the pass band of the electronic amplifier so as to attenuate any 120 cycles per sec. voltages.

Calibration System

The success of a device to count dirt is dependent on an accurate method of calibrating the entire dirt counter system. Preferably, the calibration should originate early in the system in order to catch any drift or shift in any component. It must of a certainty include any change in the light bulb. The most satisfactory method found thus far is to rotate a black radial line on a sheet of clean paper beneath the viewing system. This, of course, simulates a very large dirt speck. The black line gives a standard pulse which is much more than sufficiently high to trip all three dirt channels. This pulse is reduced to three heights by three fixed resistance networks. One merely sets the

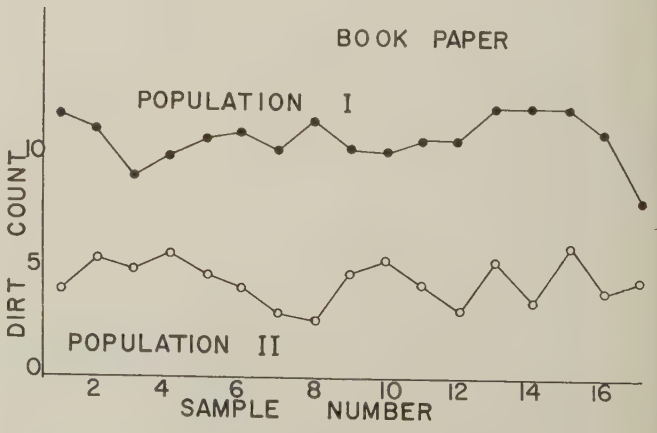

Fig. 4

discriminator level for a particular channel to just reject the pulses from the corresponding diminished standard pulse for that channel. The other channels are similarly set to just reject their corresponding artificial standard pulses in like manner. Reducing a big standard pulse in this manner has the advantage that the other smaller blemishes on the standard sheet are also reduced and, hence, less likely to be counted. Standards have been made and dipped in clear lacquer. These standards are easily kept clean by washing lightly.

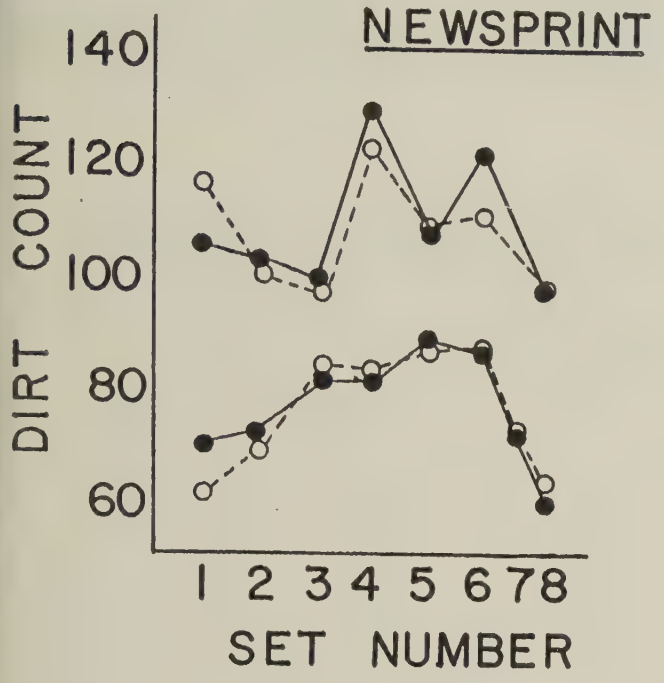

Fig. 5

The calibration system is further checked periodically by running standard populations and also by running artificially spotted standard sheets.

APPLICATIONS AS A DIRT COUNTER

The dirt counter is now used as a routine method to count dirt on bookpaper, writing paper, and newsprint. It has satisfactorily replaced the visual dirt count method.

There are, of course, many ways to evaluate the usefulness of a device to count dirt in paper. One of the early comparisons of the dirt counter to the old visual method was to compare the results from both methods on paper taken from two populations. Samples of paper were taken at random from both populations to be tested on the dirt counter. In Fig. 4 the dirt counter data are plotted for the two populations. The two populations were known to be different on the basis of the visual grading. The upper curve in Fig. 4 corresponds to the population said to be less clean based on the visual system. Hence, the dirt counter could separate these populations in much the manner that the visual system did.

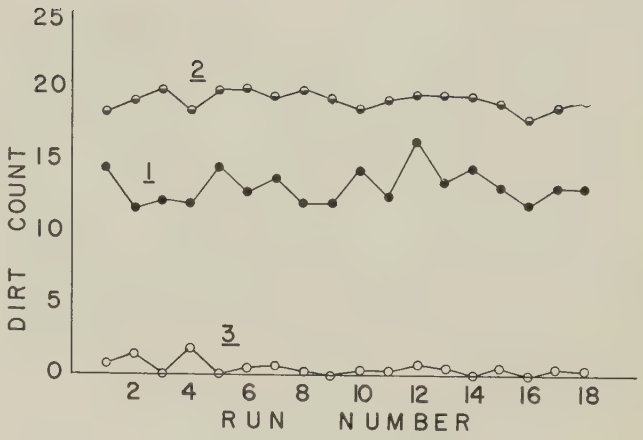

Fig. 6

Another early comparison between the dirt counter and the visual system was between two different newsprint systems known to be different in dirt count on the basis of the visual system. In Fig. 5 the dirt counter results are plotted for various sets of papers taken from the two systems. Each of the sheets was rerun later and plotted. The system known to contain more dirt by the visual method is shown as the upper two curves in Fig. 5. Thus, the dirt counter again agrees in general with the visual system.

In order to get some idea of the variability of data from the dirt counter, we ran a single sheet repeatedly over a period of one week. After each run the system was shut off and deliberately thrown completely out of calibration. The dirt counter was warmed up and

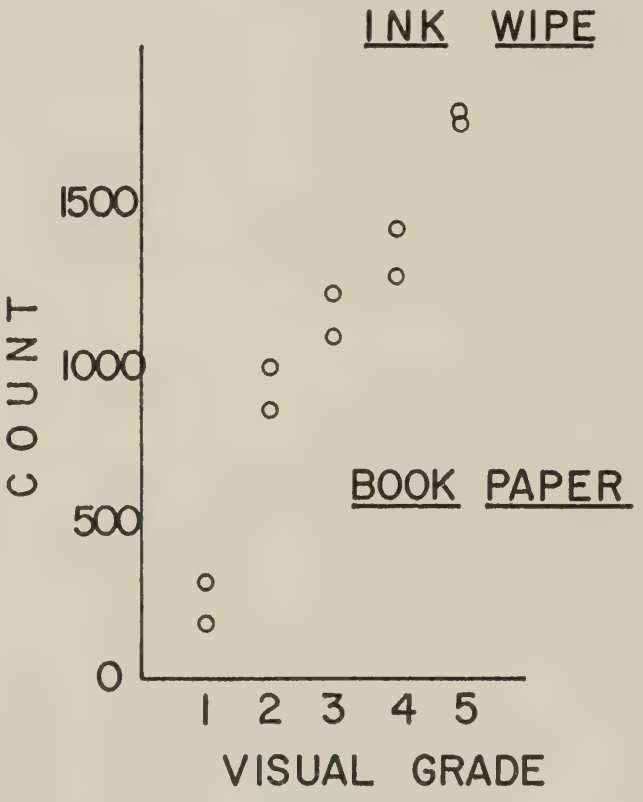

Fig. 7

calibrated before each rerun. The resulting information is shown in Fig. 6 where the dirt counter numbers are plotted against the arbitrary run number. In general, you see that the dirt counter data are fairly reproducible. One cannot, of course, expect identical results from each run because the sheet is not actually viewed in exactly the same path.

The dirt counter has other applications. It has been used to grade, or rank, samples of bookpaper that

Fig. 8

OTHER APPLICATIONS

have been ink wiped. This is an attempt to numerically evaluate the paper to give relative information on the uniformity of absorbency and roughness of the paper. In performing the ink wipe test, bookpaper samples are covered with a mixture of Donnelly no. 5 black ink and Xylol. This mixture is spread on the sheets with a drawdown bar. The sheets are then immediately wiped free of the ink with facial tissues. The resultant nonuniform samples are run on the dirt counter in the normal manner. Where the paper is absorbent, the ink strikes into the sheet and cannot be

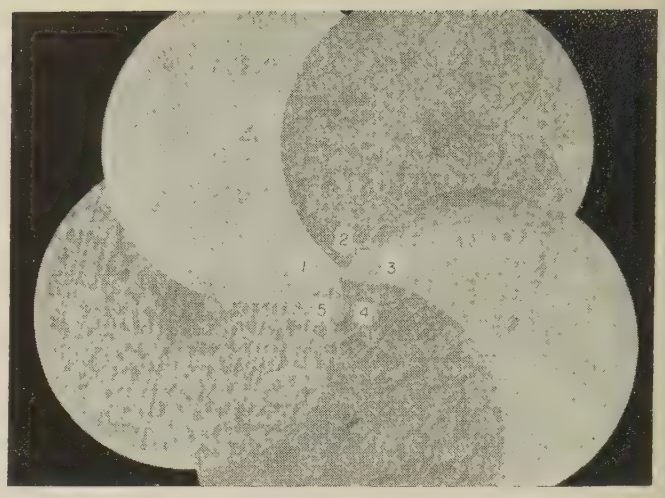

Fig. 9

rubbed off. Where the sheet is less absorbent, the ink does not strike in as fast and is rubbed off when wiped. In this manner, the absorbent and rough regions are dark in the ink wipe, and the smooth nonabsorbent regions are light in color. When these samples are run in the dirt counter, the resultant numbers are an indication of the uniformity of the ink wipe. In Figs. 7 and 8, the number appearing in the dirt counter channel no. 3 is plotted versus the visual rank or grade of the samples. In general, the dirt counter is able to separate

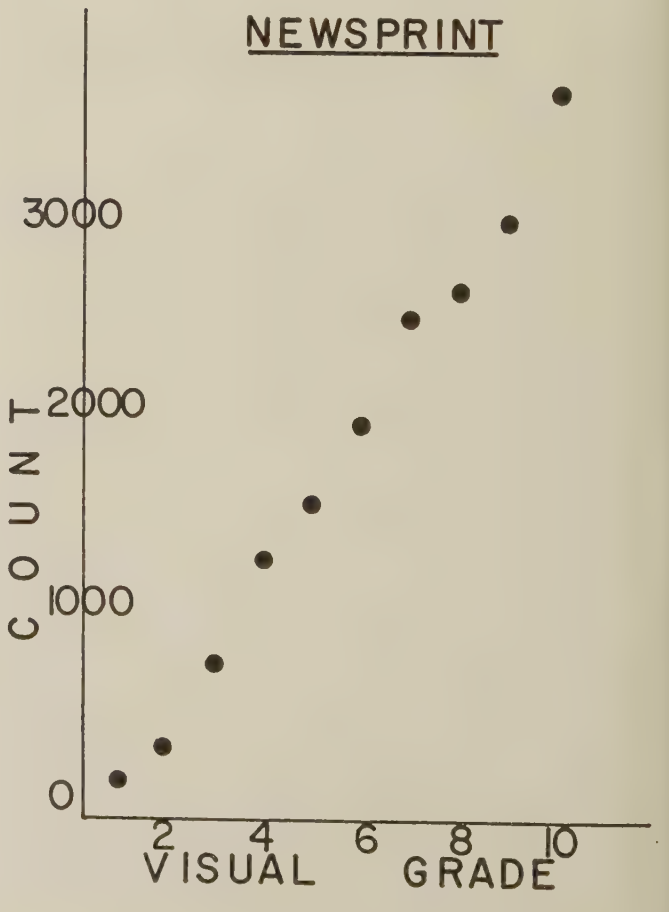

Fig. 10

the resulting ink wipe prints in much the same manner as does the visual grading.

Examples of the ink wipes are shown in Fig. 9. Notice the varied patterns that result and that the dirt counter might be required to grade were such a system used for that purpose. Some provision needs to be made for measuring the uniformity of the prints considering various areas of the nonuniformities.

Another experimental application that may prove useful in the future is to use the dirt counter to grade the uniformity of a solid letterpress print on newsprint. Sheets of newsprint of various qualities were printed at a controlled ink thickness and pressure. These solid prints were then viewed in the normal manner on the dirt counter. In Fig. 10 the number of counts in the dirt counter no. 3 channel is plotted versus the visual grade on the solid prints. Again, you see that the dirt counter separates these sheets in much the manner of the visual grader.

ACKNOWLEDGMENTS

Ralph Caston made many valuable suggestions during the course of the work. Walter Roehr suggested the possible use of the dirt counter to grade newsprint solids. Ernest Johansson did the visual grading of the ink wipe samples. The ink wipe test was developed by Harry H. Hull of R. R. Donnelly and Sons, who kindly furnished us with his ink and method.

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A Proposed Mechanism for the Acidic Chlorination of Softwood Lignin

CARLTON DENCE and KYOSTI SARKANEN

The behavior of an aqueous solution of chlorine at pH 1 toward lignin model compounds, Norway spruce lignosulfonic acid and Norway spruce wood meal was studied photometrically and by means of the isolation and identification of reaction products. Spectrophotometric results indicated that veratryl and vanillyl alcohols and spruce lignosulfonic acid alike underwent rapid substitution by chlorine into the 6-position of the aromatic nucleus. In addition, a quite general reaction was observed in which chlorine was introduced into the aromatic nucleus with accompanying replacement of the side-chain at the 1-position. The simple model carbinols such as *p*-hydroxybenzyl, vanillyl and veratryl alcohols, particularly, were susceptible to this type of replacement as were the end units of spruce lignin *in situ* which were cleaved from the remainder of the polymer chain and identified, after additional chlorination in acetic acid, as tetrachloroguaiacol. As a result of acidic chlorination in aqueous solution, the demethylation resulting in the formation of chlorocatechols and, ultimately, chlorobenzoquinones was demonstrated spectrophotometrically and by the isolation of a derivative of the former species. The significance of these various reactions is discussed in terms of the results encountered in the acidic chlorination of wood meal and various wood pulps.

The suitability of chlorine as a delignifying agent was demonstrated by Fremy and Terreil (*1*) as early as 1868 and later by Cross and Bevan (*2*) who used a procedure consisting of alternate chlorination and alkaline extraction to solubilize and remove encrustant material. In the present century these reactions have found increasing commercial application until, at present, they form the basis for one of the most important pulp purification processes, namely, the chlorination step in the multistage bleaching of pulps. As a delignification process, acidic chlorination is unique in that lignin is degraded to water- and alkali-soluble fragments under conditions which are far milder than those employed in the conventional pulping operations to remove a comparable amount of lignin. At the same time that the lignin polymer is degraded, structures are created which impart a characteristic color to the pulp. These observations indicate that several deep-seated changes result from the chlorination treatment. These transformations have been characterized only to a very limited degree although some information has been published which contributes to an understanding of the reaction mechanism. A review of the literature on this subject indicates that the following reactions may occur:

(a) A rapid introduction of chlorine into the 5- or 6-position of the guaiacyl nucleus
(b) Cleavage of the methoxyl group, the extent depending on the reaction conditions
(c) Substitution of chlorine into the aliphatic side chain
(d) Oxidation of the aliphatic and aromatic portions of the lignin structure to water-soluble fragments.

Reaction (c) may be restricted to those instances where an excess of chlorine remains after substitution at the rapidly reacting nuclear sites is complete (*3*, *4*). Kinetic studies (*5*, *6*, *7*) indicate that both a rapid and a slow reaction occur when chlorine is applied to an unbleached pulp. The former reaction, which predominates in the early stages, is thought to be a substitution reaction (a), while the other (d) is regarded as an oxidation. The reactions described above are embodied in the mechanism for acidic chlorination by Hibbert *et al.* (*8*) which is represented by Fig. 1.

The present study was undertaken in order to corroborate and supplement current theories of the

Carlton Dence, Research Associate, Empire State Paper Research Institute, and Kyosti Sarkanen, Assistant Professor of Cellulose Research, Cellulose Research Institute, State University College of Forestry at Syracuse University, Syracuse 10, N. Y.

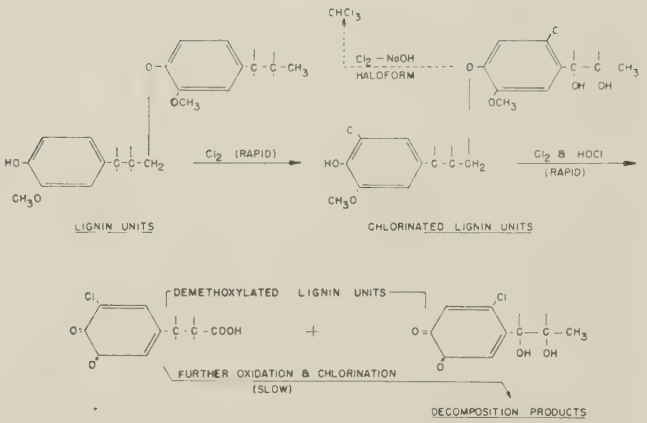

Fig. 1. Hibbert's proposed mechanism for the acidic chlorination of softwood lignin

mechanism of acidic chlorination. The approach used has been to treat various model compounds with chlorine water at pH 1 and, by means of spectrophotometric techniques and standard organic procedures, to arrive at an understanding of both the nature and sequence of the ensuing reactions. Intermediates and end products were isolated wherever possible or feasible. After obtaining the necessary information to allow an accurate interpretation of the transformations occurring during the chlorination of these simple models, lignin-like structures, and eventually lignin *in situ*, were chlorinated for comparative purposes.

PREPARATION OF MATERIALS

Norway Spruce Lignosulfonic Acid. Norway spruce wood meal (30 to 80 mesh) which previously had been extracted with alcohol-benzene solution (1:2) was subjected to a sulfite cook and subsequently treated using a slight modification of the procedure described by Kullgren (*9*). The aqueous solution of the lignosulfonic acid was stored in dark bottles under nitrogen in the cold. Analysis of the solution showed a sulfur and pentose content of 0.25 and 1.79 g.p.l., respectively. Suitable aliquots of this solution were used directly in the chlorination treatments described below.

Veratryl and Vanillyl Alcohols. The corresponding aldehydes were reduced with sodium borohydride. Veratraldehyde was prepared by methylating vanillin with dimethyl sulfate and alkali (*10*).

5-Chlorovanillyl Alcohol. Five grams of 5-chlorovanillin (*11*) was dissolved in 50 ml. of a 10% aqueous sodium hydroxide solution and treated with 1 g. of sodium borohydride. The reaction mixture was warmed for approximately 30 min. at 50 to 60°C. When reduction was complete, the pH was adjusted to 7. A white precipitate settled; this was filtered, washed with water and dried. The amount isolated was 2.4 g. or 50% of the theoretical yield of 5-chlorovanillyl alcohol which, without further purification, melted at 117 to 118°C.

ANAL. Found: C, 50.94; H, 4.72; OCH_3, 16.52; Cl, 19.89
Calculated: C, 50.94; H, 4.78; OCH_3, 16.45; Cl, 18.81

6-Chlorovanillyl Alcohol. The general procedure employed in the preparation of 5-chlorovanillyl alcohol was followed except that, in this instance, 6-chlorovanillin (*11*) was reduced. The crude alcohol melted at 161 to 165°C. and after recrystallization from chloroform containing a small amount of ethanol, at 165 to 166°C. The yield of crude material was 82%.

ANAL. Found: C, 51.36; H, 4.71; OCH_3, 16.23; Cl, 18.47
Calculated: C, 50.94; H, 4.78; OCH_3, 16.45; Cl, 18.81

5-Chloroveratryl Alcohol. 5-Chloroveratraldehyde was prepared by methylating the corresponding phenol (*11*) with dimethyl sulfate and alkali. The crude, tan-colored aldehyde melted at 58 to 59°C. Eighty-five grams of this aldehyde was dissolved in 200 ml. of methanol and allowed to react with 7 g. of sodium borohydride for 3 hr. When the carbonyl group was completely reduced, the largest part of the methanol was removed by distillation and the undistilled fraction was diluted with water prior to adjusting the pH to 7. The 5-chloroveratryl alcohol was extracted with ether and finally isolated by distilling under reduced pressure after removing the solvent. In this manner 67.2 g. (78.5% yield) of the alcohol which distilled at 132 to 134°C. under 1 to 2 mm. pressure was obtained.

ANAL. Found: C, 52.54; H, 5.50; OCH_3, 30.60; Cl, 17.17
Calculated: C, 53.54; H, 5.43; OCH_3, 30.62; Cl, 17.51

6-Chloroveratryl Alcohol. Five grams of 6-chloroveraldehyde (m.p. 145 to 146°C) was dissolved in methanol and reduced with sodium borohydride using the general procedure described for 5-chloroveratryl alcohol except that, after adjusting the pH and cooling, the alcohol precipitated as a solid; this was filtered, washed with water and dried. The yield of crude material was 73%. A fluffy, white solid melting at 82 to 83°C. was obtained after recrystallization from a chloroform-solvent naphtha mixture.

ANAL. Found: C, 53.26; H, 5.60; OCH_3, 30.60; Cl, 17.45
Calculated: C, 53.34; H, 5.43; OCH_3, 30.62; Cl, 17.51

4,5-Dichloroveratrole. This compound may be prepared by a variety of methods and several variations were employed in this investigation. With veratrole as the starting material, the theoretical amount of chlorine may be applied in its elemental form in water or as sulfuryl chloride (*12*) in a nonaqueous medium. In the former case, the veratrole is dissolved in an acetic acid-water mixture, while in the latter instance, suitable solvents are ether or chloroform. The reaction is carried out at room temperature and is complete within a few minutes. The crude product may be recrystallized from dilute ethanol as a white, granular solid melting at 83 to 84°C.

Chlorination of Norway Spruce Wood Meal. Spruce meal, previously soaked in glacial acetic acid to remove as much moisture as possible, was resuspended in glacial acetic acid and chlorinated with a stream of gaseous dry chlorine for 5 min. while agitating the slurry. The wood meal was then filtered and the filtrate rechlorinated by bubbling in additional chlorine for 45 min. while maintaining a reaction temperature of 100 to 110°C. After evaporating the solution to dryness on a steam bath, the orange-red residue was dissolved in aqueous ethanol and extracted with ether. The residue from the dried ether extract, when heated to 142 to 145°C. under 2 to 3 mm. pressure, was sublimed onto a cold "finger." The orange sublimate was removed by means of ether from which it crystallized.

These crystals were dissolved in hot aqueous ethanol and treated with a small amount of sodium hydrosulfite which destroyed the color. On cooling, a white solid precipitated which, after isolation, was found to have a melting point of 118 to 120°C. When mixed with authentic tetrachloroguaiacol (m.p. 121°C.), the melting point range was determined to be 120 to 123°C.

EXPERIMENTAL PROCEDURE

Measurement of Standard Reaction and Ionization Difference Spectra

The transformations occurring when the above materials were chlorinated were followed by measuring the accompanying spectral changes. This technique, involving the measurement of ionization and reaction difference spectra, has been applied in lignin chemistry by Aulin-Erdtman (*13, 14*) Lemon (*15*), Goldschmid (*16*) and Sarkanen and Schuerch (*17*). The spectral difference curves (ΔA curves) of the reaction mixtures resulting from chlorination were compared with standard spectral difference curves ($\Delta\epsilon$ curves) representing a single, quantitative transformation.

A standard reaction difference spectrum is the net spectral change accompanying the transformation of one compound into another. This spectrum was obtained by photometrically subtracting the spectra of equimolar solutions of the initial and final product. Solutions having concentrations of approximately 0.01 g. per 100 ml. were suitable for this measurement. Water, ethanol, or their mixtures were used as solvents, the actual choice depending on the solubility characteristics of the two compounds.

The standard ionization difference spectrum represents the spectral change occurring as a result of the ionization of an acidic group in the molecule. The acidic (phenolic) compound was dissolved in water, ethanol, or their mixtures. Two aliquots were withdrawn of such size that upon dilution the concentration would be approximately 0.004 g. per 100 ml. One aliquot was acidified with hydrochloric acid to a pH of approximately 1 to repress the ionization of the phenol and diluted to the proper volume with distilled water. The other aliquot was diluted to the same volume with 2*N* NaOH solution. The spectrum of the acid solution subtracted photometrically from that of the alkaline solution.

These optical measurements were performed using a Cary recording spectrophotometer, model 11. The instrument was standardized in such a manner that zero absorbance was obtained when the "blank" was in the light path in both reference and sample cell compartments. One-centimeter quartz cells were used throughout the investigation.

Chlorination for the Purpose of Spectral Measurement

A stock solution of the substrate was prepared at a concentration of 0.4 g.p.l. Water was used as a solvent wherever possible, otherwise aqueous acetic acid mixtures were used. A 25-ml. aliquot (equivalent to 0.01 g.) was withdrawn and placed in a 100-ml. glass stoppered volumetric flask. Dilute hydrochloric acid (0.1 *N*) was added and the solution brought to 20°C. in a constant temperature bath. The chlorine water was prepared by diluting a concentrated solution (*ca.* 6 g.p.l. chlorine) with 0.1 *N* HCl until a solution having a concentration of approximately 1.0 g.p.l. chlorine resulted. This was placed in a flask fitted with inlet and outlet tubes and the temperature adjusted to 20°C. An aliquot, providing the desired molar ratio of chlorine to substrate, was removed by displacement of the liquid by nitrogen into a pipet attached to the outlet tube, added to the hydrochloric acid-buffered solution of the substrate and allowed to react for 15 min. Any excess chlorine was destroyed by the addition of 0.1 *N* $Na_2S_2O_3$ solution. Depending on whether reaction or ionization difference spectra were to be measured, varying procedures were followed.

Where reaction difference spectra were to be measured, the chlorinated substrate solution was diluted to 100 ml. with distilled water. The blank in this case consisted of an aliquot of the stock solution of the substrate and dilute hydrochloric acid, both of the same magnitude as that used in the chlorination, diluted to 100 ml. with distilled water.

The procedure for the measurement of ionization difference spectra coincided with that described previously for standard ionization difference spectra.

The spruce lignosulfonic acid was chlorinated by the same technique as adopted for the model compounds. A 10-ml. sample of the above material was sufficient for chlorination when reaction difference spectra were to be measured. A smaller sample (5 ml. or less) proved satisfactory when ionization difference spectra were required.

The concentration of the lignosulfonic acid in the stock solution was calculated using Aulin-Erdtman's value of $\log\epsilon$ 280 mμ = 3.47 for sulfite waste liquors (*18*) and the absorbance value for the lignosulfonic acid

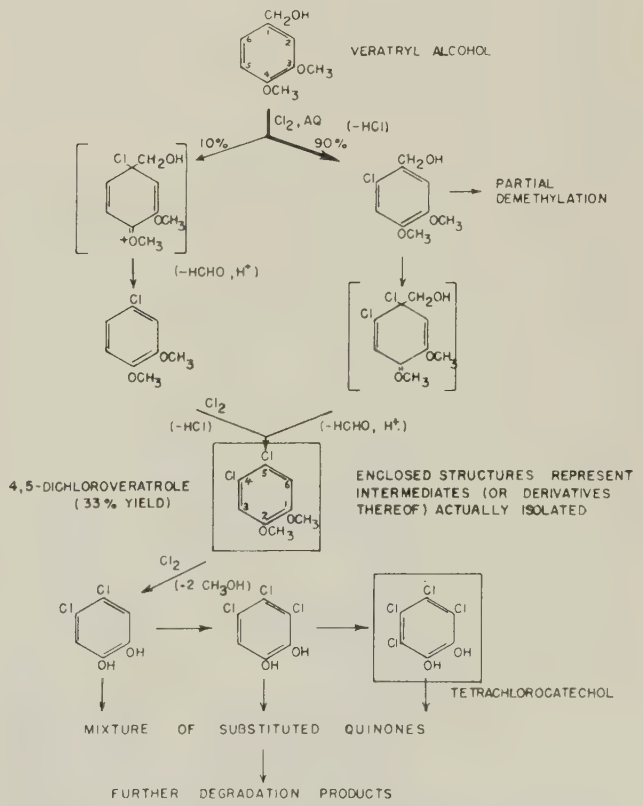

Fig. 2. Mechanism for the acidic chlorination of veratryl alcohol

solution at the same wavelength. The value for the concentration was found to be 2.1×10^{-2} moles MeO per liter. In the course of this investigtion, wherever chlorine was applied in a specified ratio to the lignosulfonic acid, calculations were based on this value.

RESULTS AND DISCUSSION

As a result of studies related to the nature of the acidic chlorination of veratryl alcohol, the authors, in a separate publication (*19*) have proposed the reaction sequence represented in Fig. 2. Because of the relation they bear to the reactions occurring during the acidic chlorination of softwood lignin, the various steps of this reaction sequence are discussed below in detail.

Aromatic Substitution Reactions at pH 1

At pH 1 the reactive halogen species present in the largest amount is molecular chlorine, although hypochlorous acid (*ca.* 4%) exists in equilibrium with it at this pH and concentration. These two species may cause either substitution or oxidation, the former process being more commonly associated with molecular chlorine and the latter with hypochlorous acid. During the chlorination of veratryl alcohol, both processes affect markedly the course and extent of the reaction.

The electronic influences operative in the veratryl nucleus during electrophilic substitution reactions, such as chlorination, are such that the electrophile is directed strongly to the position *para* to each of the methoxyl groups. When applied to veratryl alcohol, substitution *para* to the 3-methoxy group gives rise to the expected product, 6-chloroveratryl alcohol. Substitution of chlorine *para* to the 4-methoxy group, causing the displacement of the carbinol side-chain, is a reaction of particular significance in pulp bleaching and will be discussed in the next section. Substitution of chlorine into the 6-position and displacement of the carbinol group at the 1-position are competing reactions, but the former greatly predominates since its rate constant is greater by a factor of ten than that of the latter reaction. Substitution in the 5-position cannot be excluded from consideration but, inasmuch as the model 4,5-dichloroveratrole was prepared in good yield by direct chlorination of veratrole, the reaction probably takes place only to a very limited extent, if at all. By the same reasoning, substitution of chlorine into the 2-position must be negligible. The apparent absence of substitution at the 2- and 5-positions is in accordance with the observed low *ortho* to *para* ratio observed in substitutions by molecular chlorine (*20*). The decisive influence of the methoxyl group in determining the position taken by the electrophile also has been demonstrated by Strauss and Sarkanen (*21*) who chlorinated 3,4,5-trimethoxybenzyl alcohol under acidic conditions and obtained 1,2,6-trichloro-3,4,5-trimethoxybenzene.

Studies with other model substances such as vanillyl alcohol and the barium salt of 3-methoxy-4-hydroxytoluene-ω-sulfonic acid have shown that chlorine substitutes in the 6 - position of these nuclei as well. Whether this occurs to the extent noted with veratryl alcohol as yet has not been ascertained. However, substitution would be governed to a greater degree by the free phenolic hydroxyl group which would suggest that a greater amount of 5-substitution should occur.

Electrophilic Displacement of Side Chain Groups by Chlorine

The replacement of the carbinol side-chain by chlorine is a general reaction since other model compounds such as *p*-hydroxybenzyl, vanillyl, syringyl and 6-chloroveratryl alcohols were found to undergo a similar reaction. The susceptibility of the carbinol side-chain of syringyl alcohol to cleavage by chlorine is significant since it reflects the applicability of the reaction to hardwood as well as to softwood lignin systems. The fact that 6-chloroveratryl alcohol underwent this cleavage reaction indicates that a chlorine atom *ortho* to the carbinol group exerts no noticeable retarding or inhibitory effect.

Cleavage of the side-chain was retarded when the aliphatic side chain was etherified as in the cases of the methyl ether of veratryl alcohol and pinoresinol dimethyl ether. Moreover, when model substances such as γ-veratrylpropane and the barium salt of 3,4-dimethoxytoluene-ω-sulfonic acid were chlorinated, no side chain cleavage was detected as judged by the failure to isolate 4,5-dichloroveratrole from among the reaction products. On the other hand, the side chains of *p*-hydroxybenzaldehyde and *p*-hydroxybenzoic acid were replaced easily by chlorine forming 2,4,6-trichlorophenol in good yield under identical reaction conditions. Etherification of the 4-hydroxy group, therefore, appears to have a profound retarding effect on the replacement of the side chain.

Demethylation and Subsequent Reactions

The several species present in the aqueous chlorination mixture which conceivably could be responsible for, or contribute to, demethylation are molecular chlorine, hypochlorous and hydrochloric acids. Neither of the latter two substances was found capable of removing the methyl group. Consequently, molecular chlorine appears to be the reagent essential to the reaction. Qualitatively, at least, this is in accord with the conclusion of Hibbert *et al.* (*8*) who found that demethylation by chlorine occurred most easily in acid solution, less well in neutral solution and only to a negligible extent in alkaline solution. This decrease in the ease of demethylation parallels the decrease in the amount of elemental chlorine present in the system. On the basis of a study made by Strauss and Sarkanen (*21*), it appears that chlorine serves as a hydrolyzing catalyst in this reaction.

Another factor which exerts a considerable effect is the chlorinating medium itself. In preparing many of the chloroguaiacyl and chloroveratryl derivatives used in this study, the chlorinations were conducted in nonaqueous media and, although there was some indication that a small amount of demethylation had occurred, the group remained largely intact. Although most investigators have found that the presence of water even in small amounts promotes the demethylation in the presence of elemental chlorine, chlorine itself, under anhydrous conditions, may achieve the same result but perhaps by a different mechanism.

Isolation of a partially demethylated 6-chloroveratryl

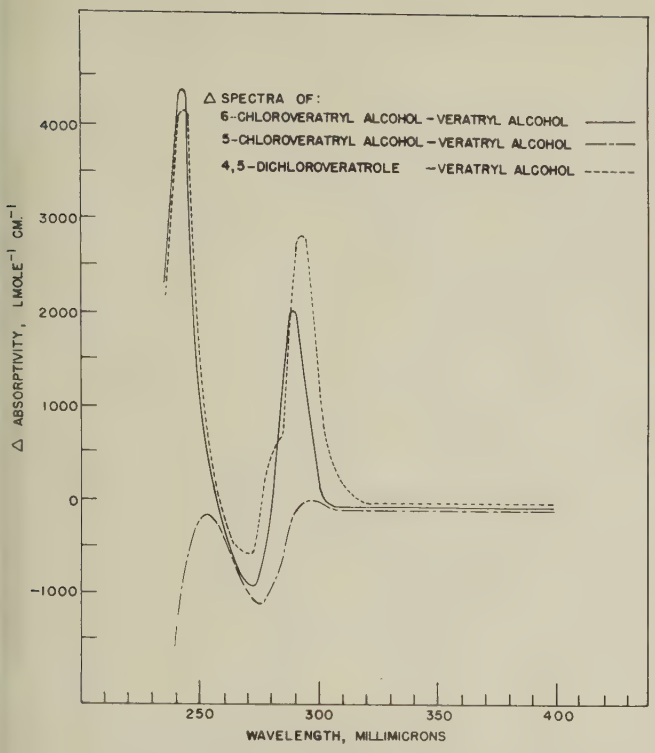

Fig. 3. Standard reaction difference spectra of 5- and 6-chloroveratryl alcohols and 4,5-dichloroveratrole

unit serves to illustrate that demethylation, although not occurring with the rapidity of the aromatic substitution of chlorine, does compete with the electrophilic displacement of the side chain.

Further substitution of 4,5-dichloroveratrole as a major reaction was deemed improbable since experience in preparing model tri- and tetrachloroveratrole indicated that far more stringent reaction conditions were required to bring about these transformations than normally were used in this study. Further chlorination of substituted catechols, however, is much more likely due to the greater reactivity imparted by the two phenolic hydroxyl groups.

The formation of substituted quinones from the corresponding catechols is in keeping with the generally recognized sensitivity of these structures to oxidants. Spectrophotometric measurements have indicated that tetrachlorocatechol is transformed to the corresponding orthobenzoquinone in the presence of a small amount of chlorine.

Reaction Difference Spectra as a Means of Following the Course of the Chlorination Reactions

Because of the heterogeneous nature of natural lignin and lignin artifacts, the isolation and identification of reaction intermediates resulting when these substances are allowed to react with chlorine is extremely difficult. A more promising approach to the problem has been made through the use of spectrophotometric techniques in which the spectral changes occurring at different stages of the reaction were compared with standard reaction difference spectra which represented the expected transformations; namely, substitution of chlorine into the 5- or 6-position of the guaiacyl or veratryl nucleus and electrophilic displacement of the carbinol side-chain by chlorine.

The standard reaction difference spectra of 5- and 6-chloroveratryl alcohols are shown in Fig. 3. The corresponding curves for 5- and 6-chlorovanillyl alcohols (not shown) have similar forms over the entire ultraviolet wavelength range. The Δ absorptivity values for the characteristic maxima of these compounds and 4,5-dichloroveratrole are shown in Table I.

The chlorinated veratryl and vanillyl alcohols possess well-defined maxima at 290 to 294mμ and at 240 to 242mμ. The spectra of the 5-chlorinated veratryl and vanillyl alcohols, on the other hand, lack well-developed maxima in the vicinity of 290mμ but possess absorption maxima at 252 and 242mμ, respectively. It is apparent from these results that the peak in the vicinity of 292mμ is indicative of chlorination in the 6-position. The corresponding peak for 5-substitution is of such low intensity as to have no practical value. The reaction difference spectrum of 4,5-dichloroveratrole is similar in form to that of 6-substituted veratryl and vanillyl alcohols although the maximum at 292mμ is broadened and intensified. Using these standard curves as a basis for comparison, various model compounds, in addition to Norway spruce lignosulfonic acid, were chlorinated and their reaction difference spectra measured.

Table I. Δ Absorptivity Values for Absorption Maxima of Standard Reaction Difference Spectra

Reaction difference spectrum	*Wavelength at absorption maximum, mμ*	*Δ Absorptivity, l/mole cm.*
5-Chloroveratryl alcohol-veratryl alcohol	252	−200
6-Chloroveratryl alcohol-veratryl alcohol	290	2090
	242	4290
4,5-Dichloroveratrole-veratryl alcohol	292	2820
	243	4220
5-Chlorovanillyl alcohol-vanillyl alcohol	242	750
6-Chlorovanillyl alcohol-vanillyl alcohol	294	2190
	240	2190

The reaction difference spectra resulting from the application of varying amounts of chlorine to veratryl alcohol are shown in Fig. 4. In all instances the narrow absorption band at approximately 290mμ denoting 6-chlorination can be observed. After 2.8 moles of chlorine were added, there was no further increase in the absorption intensity at 290mμ and additional amounts of chlorine served only to destroy the peak.

When less than one mole of chlorine was applied, there was no absorption in the wavelength range bordering on the visible spectral region illustrating that some substitution had occurred before chlorine was consumed by any side reaction such as quinone formation. However, for increasing applications up to one mole, absorption was observed in the visible region indicating that, before monosubstitution was complete, other reactions had begun to occur. There is an indication of an absorption maximum at approximately 400mμ after the addition of slightly more than one mole of chlorine. When still more chlorine was added, the absorbance in this region first grew and eventually decreased. Tentatively, this peak has been ascribed to a substituted orthobenzoquinone. The parent compound (*o*-benzoquinone) has an absorption maximum in this region the exact position and intensity of which

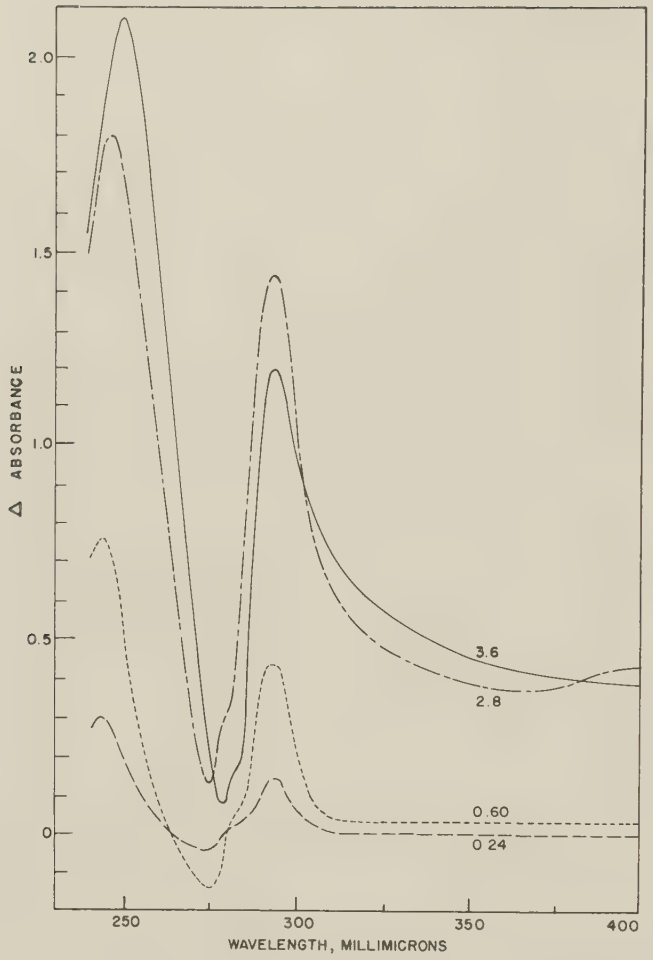

Fig. 4. Reaction difference spectra derived from the acidic chlorination of veratryl alcohol at various chlorine/substrate ratios

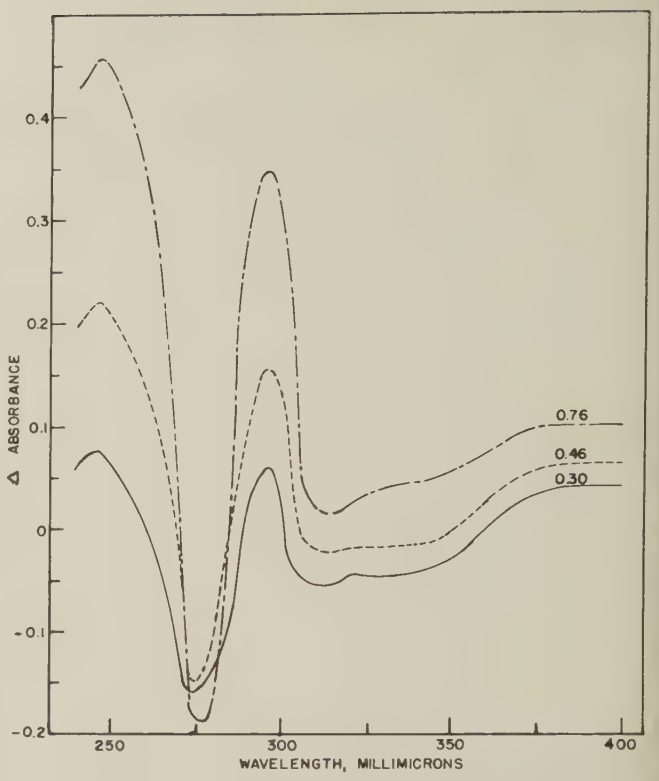

Fig. 5. Reaction difference spectra derived from the acidic chlorination of Norway spruce lignosulfonic acid at various chlorine/substrate ratios

is dependent on the solvent and the length of time the solution stood before measurement (*22*).

Vanillyl alcohol and the barium salt of 3-methoxy-4-hydroxytoluene-ω-sulfonic acid were chlorinated under the same conditions and found to be transformed to products whose reaction difference curves were similar to those of veratryl alcohol.

The spectra derived from the chlorination of spruce lignosulfonic acid are shown in Fig. 5. The absorption maximum at 294mμ which grew with increasing applications of chlorine reflects the increase in 6-substitution. The absence of isosbestic points, however, indicates that, in addition to the principal substitution reactions, one or more side reactions occur. The close similarity of these curves to those obtained when veratryl alcohol was chlorinated is immediately apparent. Substitution in the 6-position of the aromatic nucleus thus was found common to all four materials tested.

Vanillyl and veratryl alcohols and spruce lignosulfonic acid also were chlorinated at a pH of 5 where the principal reactive species is hypochlorous acid. Each of the reaction difference spectra derived from these chlorination treatments possessed a maximum at 292 to 295mμ characteristic of 6-substitution. However, whereas the spectrum derived from veratryl alcohol was nearly identical to that of the standard reaction difference spectrum for 6-substitution throughout the entire ultraviolet region, the spectral curves of vanillyl alcohol and lignosulfonic acid showed a noteworthy deviation in the 350 to 400mμ region. The increase in absorption in this region may be attributable to the formation of quinones or other chromophores resulting from oxidation by hypochlorous acid. Etherification of the phenolic hydroxyl group at the 4-position of the guaiacyl nucleus thus appears to stabilize the structure against oxidative degradation.

The Use of Ionization Difference Spectra in Following the Course of the Chlorination Reaction

The ionization of phenolic groups has been shown to result in a bathochromic shift of absorption bands toward the visible region of the spectrum (*23*). The standard ionization difference spectra of the model phenols used in this study possessed distinct differences which made it possible to classify them into three groups. A standard ionization difference spectrum representing each of the three types is shown in Fig. 6a. This classification was based on the position of the higher wavelength absorption maximum. One group, which included vanillyl (*24*), 6-chlorovanilly and 5-chlorovanillyl alcohols and the barium salt of 3-methoxy-4-hydroxytoluene-ω-sulfonic acid, showed an absorption maximum at approximately 295 to 300mμ. With a second group, composed of chloroguaiacols and chlorocatechols, the absorption maximum was found in the 305 to 315mμ range. A distinct third type is illustrated by vanillin where the absorption maximum appears at 347mμ (*16*). The absorptivities of these various compounds are presented in Table II. The spectra of the chlorocatechols were found to change on standing in solution so that the spectral measurements were performed immediately after the compound was dissolved. Reasonably good reproducibility of Δ

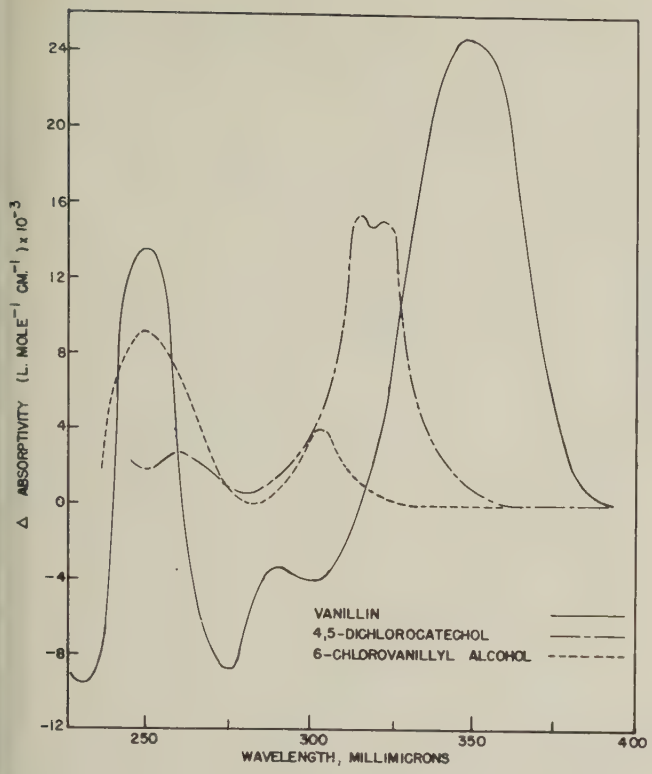

Fig. 6a. Standard ionization difference spectra of various model phenols

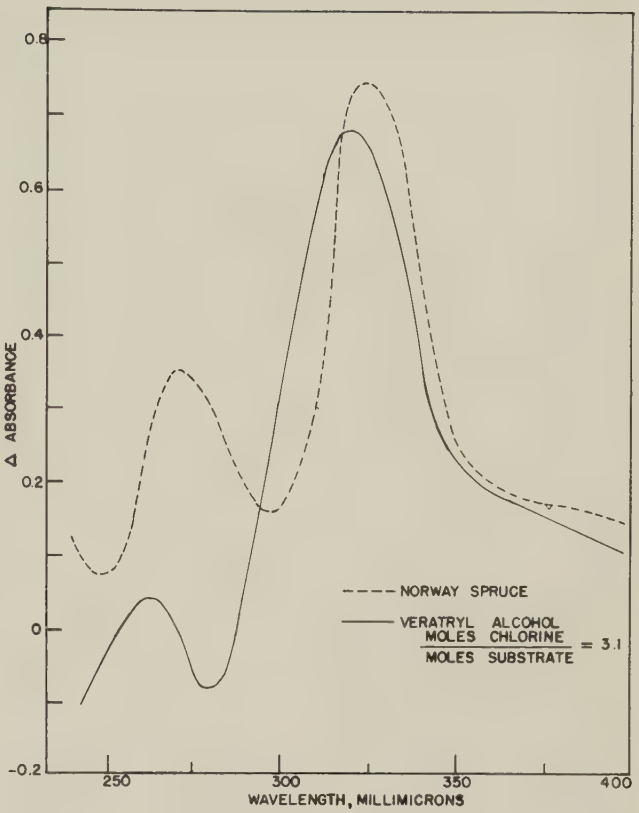

Fig. 6b. Ionization difference spectra derived from the acidic chlorination of veratryl alcohol and the aqueous extract of chlorinated spruce wood

absorptivity values was achieved for the higher wavelength maximum of each spectrum, but not for the lower. For this reason, Δ absorptivity figures of the latter were not reported.

Table II. Δ Absorptivity Values for Absorption Maxima of Standard Ionization Difference Spectra

Compound	*Wavelength at absorption maximum, mμ*	*Δ Absorptivity, l/mole cm.*
Type I		
Vanillyl alcohol (*24*)	292	4220
	249	9800
6-Chlorovanillyl alcohol	302	4190
	254	8540
5-Chlorovanillyl alcohol	297	4310
	252	8900
Barium salt of 3-methoxy-4-hydroxytoluene-ω-sulfonic acid	295	4390
	250	11500
Type II		
Guaiacol (*16*)	290	3300
	239	7900
4,5-Dichloroguaiacol	308	4590
	250	7940
4,5,6-Trichloroguaiacol	310	4490
	253	8530
Tetrachloroguaiacol	315	4530
	252	9160
Catechol (*25*)	298	5130
	253-4	5250
4,5-Dichlorocatechol	315	14430
4,5,6-Trichlorocatechol	315	4540
Tetrachlorocatechol	312	3240
Type III		
Vanillin (*16*)	347	25600
	249	9700

The ionization difference spectrum of vanillin illustrates the circumstance where the phenolic hydroxyl group is conjugated through the ring with an alpha carbonyl group. The maximum in the vicinity of 350mμ appears to be characteristic for compounds possessing this structural element (*26, 27, 28*). The position of the absorption maximum in the 295 to 350mμ range of the spectrum thus is seen to give some indication of the structure of the absorbing unit.

Chlorination of veratryl alcohol and subsequent measurement of the ionization difference spectrum (Fig. 6b) of the reaction mixture indicated that an ionizable group had been formed. The absorption maximum occurred at approximately 320mμ which suggested that the absorbing material was a chlorocatechol rather than a chloroguaiacol. Moreover, subsequent analysis of residues of the acidic material showed that removal of the methyl group virtually was complete after the reaction so that the spectrum undoubtedly was that of a substituted catechol rather than of a guaiacol. The intensity of the absorption maximum grew with increasing application of chlorine up to a certain limit when further addition caused it to diminish. This point may have marked the beginning of the formation of significant amounts of tri- and tetrachlorocatechol each of which has a substantially lower absorptivity than dichlorocatechol in this wavelength region.

The ionization difference spectrum of an aqueous extract of chlorinated Norway spruce wood also is shown in Fig. 6b. The strong resemblance to the ionization difference spectrum of dichlorocatechol is immediately apparent. Whether or not this represents the spectrum of monomer units or of larger demethylated fragments is not known. The fact that the absorbing material was water-soluble and not extractable with ether points to a hydrophilic structure of reasonably low molecular weight.

The ionization difference spectra resulting from the chlorination treatment, as well as the spectrum of un-

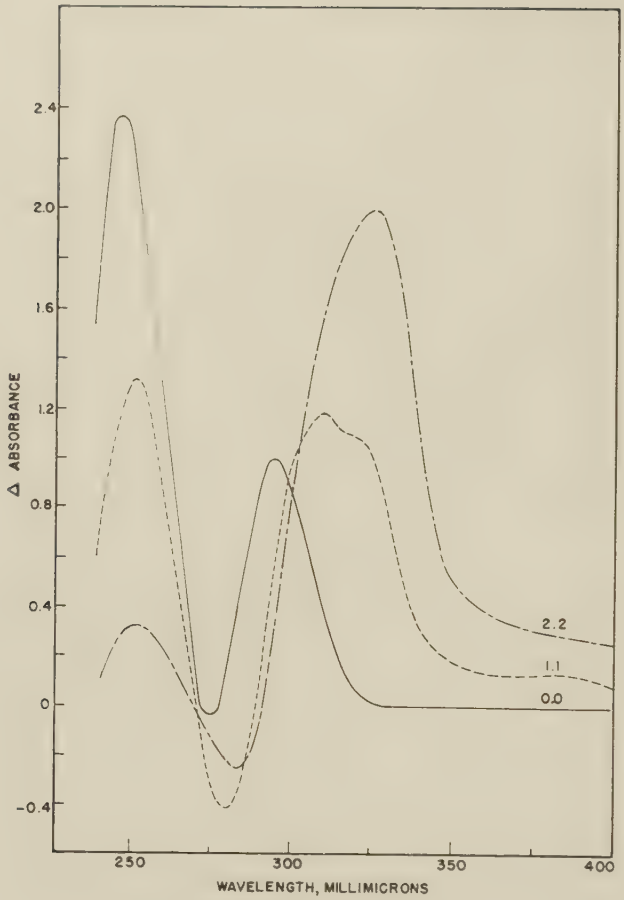

Fig. 6c. Ionization difference spectra derived from the acidic chlorination of vanillyl alcohol at various chlorine/substrate ratios

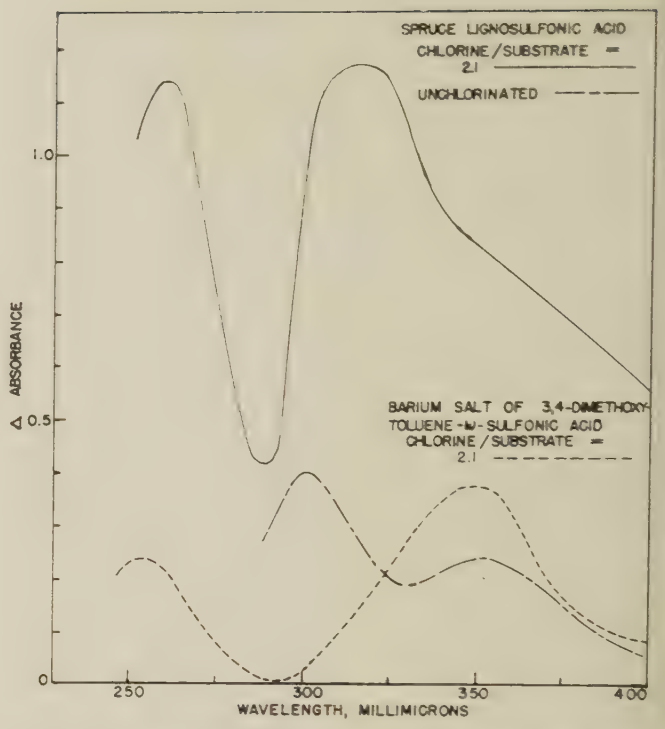

Fig. 6d. Ionization difference spectra derived from chlorinated and unchlorinated spruce lignosulfonic acid and the chlorinated barium salt of 3,4-dimethoxytoluene-ω-sulfonic acid

treated vanillyl alcohol, are shown in Fig. 6c. The spectral changes observed in this figure suggest various stages in the transformation of the original alcohol to a substituted catechol. In general, on the basis of their ionization difference spectra, both vanillyl and veratryl alcohols undergo a similar series of reactions with chlorine, but vanillyl alcohol is more reactive and reaches the quinone stage at a lower chlorine to substrate ratio. This serves as another indication that when the phenolic hydroxyl groups are completely etherified, the nucleus is stabilized against degradative attack.

The ionization difference spectra derived from the acidic chlorination of the barium salt of 3,4-dimethoxytoluene-ω-sulfonic acid and spruce lignosulfonic acid, together with the original spectrum of the latter, are shown in Fig. 6d. The form of the spectrum of the model sulfonate after chlorination was in marked contrast to that of vanillyl or veratryl alcohols. The absorption maximum at 350mμ grew with increasing application of chlorine up to a point and then decreased. The position of the maximum points to a phenolic hydroxyl group conjugated through the ring with an alpha carbonyl group. The intensity of this maximum is far less than is normally found for the *p*-hydroxybenzoyl structure, leading to the conclusion that either there is incomplete conversion to this species or that, once having formed, it is rapidly degraded. The intense color developed in the early stages of the chlorination reaction suggests that degradation proceeded through the catechol to the quinone stage.

Since it is probable that the benzyl alcohol groups were incompletely sulfonated by the cooking liquor, the lignosulfonic acid solution should have behaved toward chlorine as a mixture of the corresponding sulfonates of veratryl and vanillyl alcohols and the corresponding unsulfonated alcohols. In this regard it will be observed that the untreated sulfonic acid has the anticipated absorption maximum in the 300mμ region. Application of slightly more than one mole of chlorine shifted the spectrum of the untreated material toward the visible region, probably as a result of nuclear substitution. No definite conclusion can be drawn about the maximum at 360mμ since the starting material had a similar peak. By analogy with the model sulfonate, however, some of this absorbance could be attributed to the formation of carbonyl groups at the side-chain. Addition of 2.1 moles of chlorine completed the metamorphosis and the spectrum assumed the contours of a chlorocatechol.

Isolation of Tetrachloroguaiacol from Chlorinated Spruce Wood

Among the various structural elements known to occur in softwood lignin are hydroxyl groups situated on the side chain carbon atom alpha to the guaiacyl nucleus. To the extent that these carbinol groups occur in lignin, breakage of the bond between the alpha carbon atom and guaiacyl nucleus with concurrent chlorine substitution was anticipated. This was borne out experimentally when a lignin end unit was isolated from among the chlorinated reaction products as tetrachloroguaiacol. The chlorination was carried out in glacial acetic acid to preserve the methoxyl group intact, a precaution which is necessary since its removal renders the ring sensitive to further degradative attack. Figure 7 represents the transformations which are believed to occur in the reaction. The degree of substitution in the isolated product is without significance since the chlorination was carried to the point where the ring was completely substituted so as to eliminate the prob-

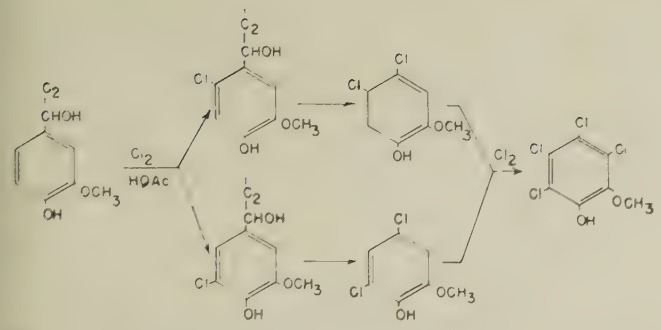

Fig. 7. Reaction sequence for the isolation of a softwood lignin end unit as tetrachloroguaiacol

lem of having to separate and identify isomers.

In this connection it is of interest to note that Cross and Bevan (*2*) isolated a chloroquinone by subliming chlorinated jute lignin. The reduced quinone was identified as trichloropyrogallol.

Proposed Mechanism for the Chlorination of Norway Spruce Wood Meal

In the proposed mechanism shown in Fig. 8, the aliphatic side-chains, through which the aromatic nuclei are joined, are represented in the simplest manner possible. This was done for purpose of simplification recognizing that linkage of the β or γ carbon atoms of the side chain with the 5-carbon atom of the adjoining ring, as well as other structural elements believed to occur in lignin, may alter the course of the reactions discussed in connection with the chlorination of veratryl alcohol.

By analogy with the behavior of spruce lignosulfonic acid toward chlorine, substitution logically would be expected to take place in the 6-position except where the phenolic hydroxyl groups in the 4-position were unetherified in which case 5-substitution also would be a strong possibility. Wherever etherified or unetherified benzyl alcohol groups were present, chlorine has been shown as breaking the bond between the benzene nucleus and the aliphatic side-chain. In keeping with the fact that formaldehyde was formed when the analogous reaction took place with veratryl alcohol, the end carbon of the cleaved aliphatic side-chain has been represented as an aldehyde group. Whether or not the phenolic ether linkages at the 4-carbon atom are cleaved during the chlorination, as methoxyl groups are, was not established in this investigation. As indicated in Fig. 8, not all the phenylpropane units are cleaved so that, depending on the number and distribution of cleaved linkages and the amount of chlorine applied, a wide distribution of various size fragments would be predicted. That no simple molecular species greatly predominated was evident from the nature of the residue remaining after evaporation of the aqueous extract of the chlorinated wood, for only a small fraction of the total residue could be sublimed in vacuum.

All indication point to the conclusion that the aromatic nucleus is not highly substituted as a result of the conditions employed in the chlorination treatment. The ionization difference spectrum of the aqueous extract of the chlorinated wood which strongly resembled the spectrum of 4,5-dichlorocatechol, is supporting evidence as is the analytical data furnished by various investigators (*29, 30, 31, 32*) for the per cent chlorine in

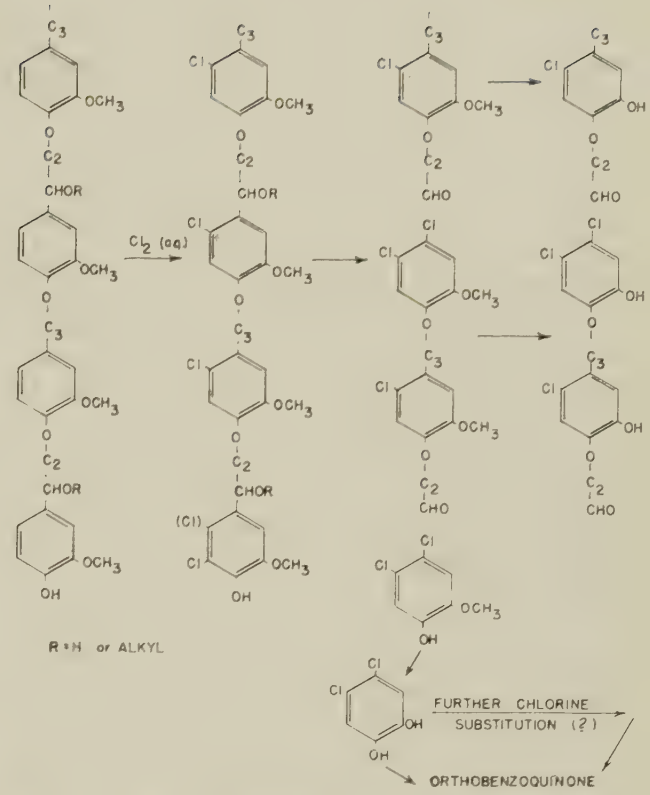

Fig. 8. Proposed mechanism for the acidic chlorination of softwood lignin

an isolated chlorolignin. The magnitude of these figures is such that, assuming a uniformly substituted product, tri- or tetrachlorinated nuclei definitely were excluded as possibilities.

The proposed mechanism is in agreement with some details of the Hibbert mechanism and at variance with others. Common to each is the initial, rapid substitution of chlorine into the guaiacyl nucleus. Hibbert proposed an oxidative degradation of the lignin polymer in which an ether linkage at the γ-carbon was cleaved and a carboxyl group formed whereas the present study has indicated that degradation occurs at the carbon atom alpha to the guaiacyl nucleus, the side-chain being replaced by chlorine. Both studies agree that demethylation occurs, but differ on the nature of the aromatic moiety resulting from the cleavage. The results of this investigation strongly suggest that demethylation occurs by a non oxidative mechanism.

The Significance of Acidic Chlorination and Demethylation to Bleaching

As a result of an acidic chlorination treatment, a considerable amount of the lignin in pulp becomes water-soluble and a still greater amount, alkali-soluble. Substitution of chlorine into the aromatic nucleus of simple organic compounds is generally accepted as being accompanied by a decrease in water-solubility so that, reasoning by analogy, chlorolignin resulting from simple substitution should have undergone a similar change in this property. The opposite effect actually observed in the bleaching of pulp can be ascribed to the offsetting side-chain cleavage and demethylation reactions. The lignin polymer chains are thus reduced to small water- and alkali-soluble fragments as this study has shown. The extent to which such reactions occur has not been elucidated completely. However, it is evi-

dent that complete scission of splitting of methoxyl is not necessary to effect the desired solubilization. The catechols arising from the cleavage of methoxyl groups can be regarded as precursors of quinones which generally have been considered as the source of the pulp color observed in the acidic bleach treatment.

It may be pointed out that the side-chain displacement is formally a substitution reaction, since one mole of hydrochloric acid is liberated for each mole of chlorine consumed. The proposed mechanism, therefore, is in agreement with the results of Giertz (*6*) on the chlorination of sulfite pulps. Contrary to earlier views, however, ordinary aromatic substitution appears to do little to aid solubilization of the residual lignin.

SUMMARY

The standard reaction and ionization difference spectra of a number of model compounds served as a basis for evaluating the structural changes which occurred in the various lignin model compounds as a result of treatment at diverse pH levels. Substitution of chlorine into the 6-position of the aromatic nucleus was demonstrated clearly in each instance when chlorine was applied to veratryl and vanillyl alcohols and spruce lignosulfonic acid at pH 1 and pH 5. Information as to the probability of 5-substitution could not be derived from the spectra. There was spectral evidence that simple nuclear substitution was retarded as the pH was increased from 1 to 5 whereas the oxidative phase of the reaction became more pronounced. The position of the absorption maximum observed in the near-ultraviolet region of the spectra was compatible with that of substituted orthobenzoquinones. Based on the behavior of veratryl alcohol toward chlorine at pH 1, substitution preceded formation of quinonoid structures. Where either or both of the phenolic hydroxyl groups were unetherified, the interim between these reactions may be shortened considerably.

The removal of the side-chain from the aromatic nucleus as a result of acidic chlorination was shown to be a general phenomenon. The ionic nature of the mechanism proposed for this reaction was based on a combination of direct experimental evidence and on a consideration of established mechanisms for similar reactions. The extent to which this scission occurred was dependent on the nature of the side-chain but, more fundamentally, it may be contingent upon the stability of the carbonium ion ejected from the nucleus. Confirmation of the applicability of the side-chain cleavage reaction to a complex system was realized when an end unit from lignin *in situ* was isolated as tetrachloroguaiacol. This has led to the hypothesis that the solubilization of lignin effected during an acidic bleach treatment may be due in part to a reaction of this type. However, as a result of the pulping treatment, the functional groups attached to the carbon atom alpha to the aromatic nucleus are altered so that the extent of the response of the alpha carbon toward cleavage will depend largely on the nature of transformation.

The ionization difference spectra revealed that, as a consequence of the acidic chlorination treatment, free phenolic hydroxyl groups were formed as intermediates in the overall decomposition process. The substituted catechols thus formed are regarded as the intermediates through which the reaction leading to substituted orthobenzoquinones takes place. The mechanism of demethylation, although as yet not completely defined, is ionic in nature and catalytically influenced by the presence of elemental chlorine and an aqueous system. Although a somewhat slower process than simple substitution, demethylation becomes a significant factor before one mole of chlorine has substituted completely in the nucleus.

The formation of free hydroxyl groups is compatible with observed behavior of pulp toward chlorine during an acidic chlorination in that the phenolic substances have enhanced water solubility due to salt formation with the alkali in the ensuing caustic extraction stage. The general reactivity of the substituted catechols acts to the detriment of the operation in one respect, however, in that chlorine is consumed oxidatively in degrading still further structures which already may have acquired sufficient water solubility to be removed.

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ASSOCIATION NEWS AND EVENTS

National Conferences

Forty-Fifth Annual Meeting, Hotel Commodore, New York, N. Y., Feb. 22–25, 1960.

Eleventh Coating Conference, Edgewater Beach Hotel, Chicago, Ill., May 23–25, 1960.

Pulp Bleaching Conference, Edgewater Beach Hotel, Chicago, Ill., June 14–16, 1960.

Fourteenth Alkaline Pulping Conference, Multnomah Hotel, Portland, Ore., Aug. 22–24, 1960.

Forest Biology Conference, Seattle, Wash., Aug. 24–27, 1960.

Eleventh Testing Conference, Pantlind Hotel, Grand Rapids, Mich., Sept. 27–29, 1960.

Tenth Corrugated Containers Conference, Royal York Hotel, Toronto, Canada, Oct. 10–13, 1960.

Fifteenth Plastics-Paper Conference, Hotel Syracuse, Syracuse, N. Y., Oct. 17–19, 1960.

Fifteenth Engineering Conference, Robert Meyer Hotel, Jacksonville, Fla., Oct. 24–28, 1960.

Local Section Meetings

Pacific: April 15, 1960, Everett, Wash., Shibley Award Meeting; May 5–7, 1960, Bellingham, Wash., or Harrison Hot Springs, Annual Meeting; September, 1960, Salem, Ore., "Modernization."

Golden Gate District (Pacific Section): Jan. 26, 1960, Visit to California Forest Products Laboratory, 3:30–4:00 p.m., Dinner at Hotel Claremont 6:00 p.m.; Mar. 29, 1960, "Recent Advances in Materials Handling," 4:00 p.m. mill visit to Nulaid plant, dinner at Cottage Restaurant, San Leandro 6:00 p.m.; May 10, 1960, True Memorial Lecture, Hotel Claremont, Berkeley.

Lakes States: March 8, 1960, mill visit Bergstrom Paper Co., Neenah; dinner at Menasha Elks Club. Theme: "Statistical Methods Applications in the Paper Industry"; April 12, 1960, dinner location to be announced. Theme: C. J. West Awards. May, social meeting—date to be announced.

Delaware Valley: Jan. 21, 1960, Engineers Club, Philadelphia, Pa., Operations Research Panel; March 26, 1960, Benjamin Franklin Hotel, Philadelphia, Pa., 1960 Philadelphia Graphic Arts Conference; April 28, 1960, Engineers Club, Philadelphia, Pa., E. J. Albert Award Program; May 27, 1960, Beliot Eastern, Downingtown, Pa., tour and outing.

Kalamazoo Valley: Jan. 21, 1960, Harris Hotel, Kalamazoo, Papermakers' Get-Together; March 17, 1960, Inman's Restaurant, Galesburg, "Graphic Arts"; April 21, 1960, Inman's Restaurant, Galesburg, "Stock Preparation"; May 12, 1960, Harris Hotel, Kalamazoo, Recognition Night. WMU Awards; June 21, 1960, Annual Golf Outing, Gull Lake Country Club.

New England: April 8, 1960, Management Night; June 3–4, 1960, Chatham Bars Inn, Chatham, Mass., Spring Meeting.

New England (Eastern District): March 14, 1960, Armand's Restaurant, Route 9, Framingham, Mass.

Ohio: Feb. 11, 1960, Colony Restaurant, Swifton Center, Cincinnati, Ohio, plant tour through Formica, a division of American Cyanamid; March 15, 1960, Manchester Hotel, Middletown, Ohio, "Two-Phase Flow in a Wet Web." Speaker: Joe Parker, Beloit Iron Works; April 12, 1960, Miami University, Oxford, Ohio, a tour of the new Paper School. Subject: "High Polymers." Speaker: H. E. Whitacre; May 12, 1960, Hartwell Country Club, Cincinnati, Ohio, Ladies night.

Ohio (Indiana District): Jan. 19, 1960, Marott Hotel, Indianapolis, Ind., "Coating," Speaker: William H. Aiken, Diamond National Corp.; Feb. 16, 1960, Plant tour to be announced; March 15, 1960, Marott Hotel, Indianapolis, Ind. Panel Discussion, To be announced; April 19, 1960, Plant tour to be announced, "Coating," Brage Golding, Purdue University; May 17, 1960, Marott Hotel, Indianapolis, Ind., "Testing," Arthur Dreshfield, The Glidden Co.

Empire State: June 10–11, 1960, Whiteface Inn, Whiteface, N. Y.

Empire State (Metropolitan District): 465 Lexington Ave. New York, N. Y. March 8, 1960, "Coating"- April 12, 1960, "Packaging"; May 13, 1960, Ladies' Night.

Empire State (Northern District): Woodruff Hotel, Watertown, N. Y. Feb. 11, 1960, "Color"; March 9, 1960, "Strength Developments in Paper"; April 14, 1960, Junior Award Night; May 12, 1960, Annual Ladies Night.

Empire State (Central District): University Club, 431 E. Fayette St., Syracuse, N. Y.

Empire State (Western District): Prospect House, Niagara Falls, N. Y.

Empire State (Eastern District): Queensbury Hotel, Glens Falls, N. Y. Jan. 21, 1960, Executives Night; Feb. 18, 1960, "Synthetic Fibers" (tentative); March 17, 1960, Junior Awards Contest; April 21, 1960, Symposium, "Wet End Machine Operation"; May 21, 1960, Annual Ladies' Night.

Maine-New Hampshire: June 17–18, 1960, Mountain View House, Whitefield, N. H., Annual Meeting.

Chicago: Chicago Bar Association, 29 S. LaSalle St., Chicago, Ill. Jan. 5, 1960, "Webfed Offset Printing"; Feb. 2, 1960, "Adhesives"; March 1, 1960, "Folding Cartons"; April 5, 1960, "Corrugating"; May 3, 1960.

Lake Erie: Jan. 21, 1960, Cleveland Engineering and Scientific Center, Conference on Pulp and Paper Testing Equipment and Methods Including Laboratory Coating and Sheetmaking Techniques; Feb. 18, 1960, Colton Chemical Co., 6626 Union Ave., Cleveland, Ohio, plant tour; March 17, 1960, Cleveland Engineering and Scientific Center, "Off-Machine Coating with Emphasis on Waxing and Barrier Types and Base Paper Requirements."

Southeastern: Jan. 22–23, 1960, Jacksonville, Fla.; March 18–19, 1960, Columbia, S. C.; May 20–21, 1960, Savannah, Ga.

Gulf Coast: Jan. 29, 1960, Stafford Hotel, Tuscaloosa, Ala.; March 18, 1960, Core Hotel, Panama City, Fla.; May 13, 1960, New Orleans, La.

Delaware Valley

The Delaware Valley Section announces "The 1960 Philadelphia Conference of the Graphic Arts Industry" to be held on Saturday, March 26, 1960, at the Benjamin Franklin Hotel in Philadelphia from 8 a.m. to 5 p.m.

The Saturday date has been selected on recommendation from representatives of the Philadelphia Litho Club and the Philadelphia Printing Ink Makers Products Club for the convenience of their members to attend.

This will be the third consecutive Annual Graphic Arts

Conference sponsored by the Delaware Valley Section and will be the first time a full day's program has been planned.

Enthusiastic reactions to the previous two conferences indicates that this bigger and better conference will also be successful and point the way to further growth and a regular annual event of far reaching interest.

Since the Philadelphia area is generally accepted as the birthplace of both the printing and papermaking industries in this country, and continues to be an important center for both these industries, it is an ideal location for holding these conferences.

Annual Meeting Personnel Service

To assist companies recruiting technical men in publicizing their personnel needs, a "Personnel Service" room will be provided at the Hotel Commodore during the 1960 Annual Meeting. Company members of the Association may post descriptions of their openings in this room which will be available to all delegates. This service was initiated at the 1959 meeting and is being repeated at the request of the companies which participated.

Interested corporate and sustaining members are asked to forward brief position descriptions to the Association office by Feb. 15, 1960. These descriptions which will be displayed on bulletin boards in the "Personnel Service" room on Monday through Wednesday, February 22–24. These descriptions should include the name and address of a company representative who may be approached if additional information or an interview is desired. Companies planning to have a personnel representative at the Annual Meeting may add his name and hotel room number to the descriptions after he has arrived in New York. If the position descriptions of any one company cover more than a single sheet, the subsequent pages will be posted behind the first sheet on the bulletin boards.

The Association's role in this activity is expressly limited to providing the area to its company members without charge, supplying an attendant, and publicizing the location of the room in the program booklet and on the conference bulletin boards.

Other Associations

Verein der Zellstoff- und Papier-Chemiker und-Ingeniere, Garmisch-Partenkirchen, Germany, June 28–July 1, 1960.

The Twenty-Third Annual Short Course for Superintendents and Operators of Water and Sewerage Systems will be held at Louisiana State University, Baton Rouge, La., on March 16–18, 1960.

High-Speed Testing Symposium

The second annual Symposium on High-Speed Testing will be held at the Hotel Somerset, Boston, Mass., Jan. 27, 1960. The program of the meeting is as follows:

1. "New High-Speed Tensile Tester for Polymer Films," Gordon D. Patterson, Jr. (Du Pont).
2. "Smokless Powder Testing of Polyurethane Foams at Rates of from 1 to 10^5 Inches per Inch per Minute," by John W. Jones (Du Pont).
3. "Response of Elastomers, Over a Wide Frequency Range, to a Variety of Forcing Functions," by Martin Berger (Esso).
4. "High-Speed Testing of Solid Propellants," by K. H. Sweeney and Kenneth Bills (Aerojet-General).
5. "Problems Associated with Strain Measurements of Textiles under Impact Loading Conditions," by Stanley Backer and Jan Rizek (MIT).
6. "Critical Comparison of Moving Bar Impact with Gas Expansion Machines for Dynamic Loading of Metals," by Joseph M. Kraft (U. S. Naval Research Laboratory).

The program has been prepared under the co-chairmanship of Professor A. G. H. Dietz, MIT, and F. R. Eirich, Polytechnic Institute of Brooklyn. The Symposium is sponsored by the Plas-Tech Equipment Co., Natick, Mass.

Italian Translations of TAPPI Standards

The recently chartered Italian Section has undertaken the translation of TAPPI Standards into Italian. It is expected that several of these will be translated each month and published in Italy for distribution to Italian TAPPI members. Translations of Standard Methods T 213, T 400, T 402, T 403, and T 404 have already been distributed.

Association members living outside of Italy may obtain copies of all translations at a nominal cost. Inquiries should be directed to Dr. Piero Bersano, Cartiere Burgo, Turin, Italy.

Erratum

In the paper "Animal-Glue Testing for Paper Sizing," by P. B. Davidson and H. B. Bodenhagen (*Tappi* 42, No. 9: 720–723 (Sept., 1959)) wrong headings were used in that it was originally proposed to publish it in two parts. Part II should be under the heading "Results of Test Method" on page 728.

Testing Equipment Suppliers

The suppliers listed below have indicated their willingness to certify to any individual purchaser, upon request, that the instruments or material listed under their names do actually comply with the requirements of the TAPPI Standards and Suggested Methods for which they are listed, at the time of certification.

Suppliers of testing equipment are invited to examine the list in order to determine whether their equipment is included. Forms for certification of additional equipment may be obtained from A. J. Winchester, TAPPI, 360 Lexington Ave., New York 17, N. Y. The list will be republished periodically.

Method	*Apparatus*	*Suppliers*
T 16 m-54 Sieve Analysis of Pulpwood Chips	Sieve, screen mechanism, balance, accessories	TMI WAC
T 200 m-45 Laboratory Processing of Pulp (Beater Method)	Disintegrator, beater	TMI VIW
T 202 Chlorine Consumption of Pulp	Koerner-type mill or paper defibrator	AEC
T 205 m-58 Forming Handsheets for Physical Tests of Pulp	Disintegrator, sheet machine with stirrer, couch roll, couch plate, pump and press with pressure gage, press template, disks, drying rings, blotting paper, cup, bucket, graduated cylinder	TMI
T 205-m Forming and Testing Pulp Sheets	Press, pump, disintegrator, sheet machine, stirrer, couch roll, couch plates, drying rings, disks	HMC
T 206 m-55 Cuprammonium Disperse Viscosity of Pulp	Viscosity tube	TMI EHS WC
T 210 m-45 Weighing, Sampling and Testing of Wood Pulp for Moisture	Oven	WAC
T 211 m-54 Ash in Pulp	Crucible, analytical balance, desiccator, electric muffle furnace, drying oven	TMI
T 215 m-50 Copper Number of Pulp	Grinder (Koerner type) steam (or oil) bath	TMI

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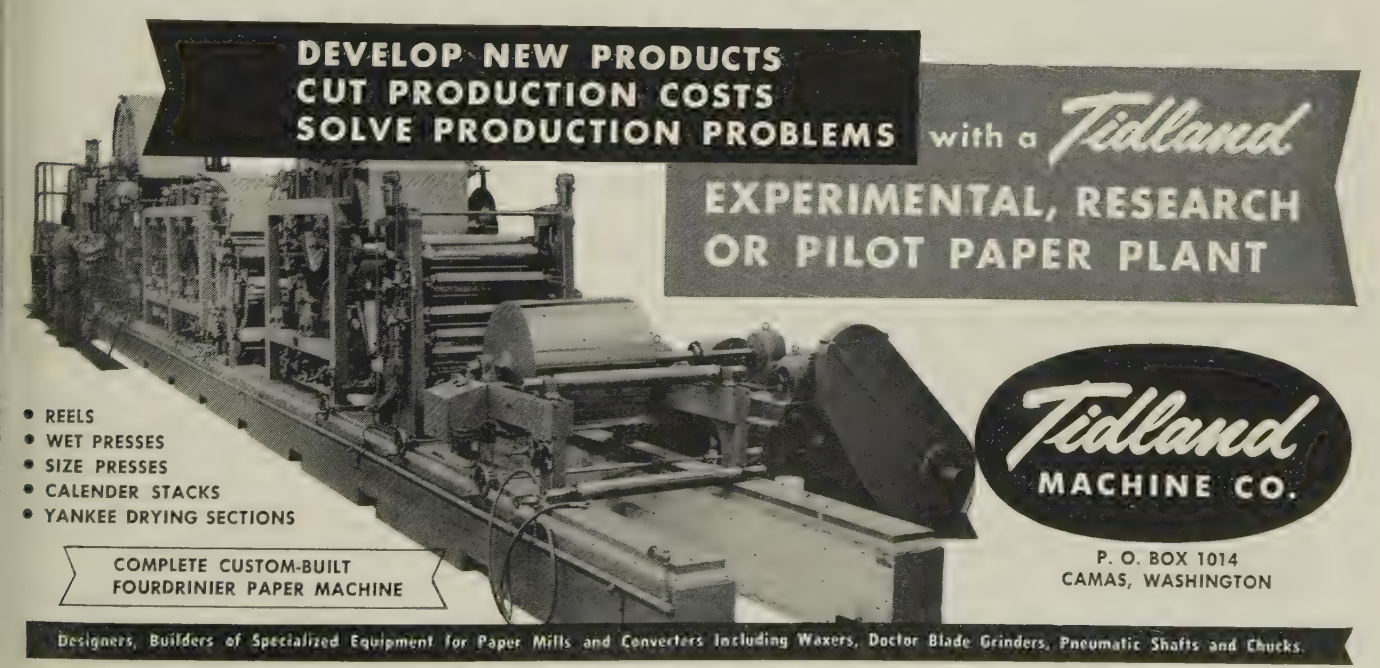
Method	Apparatus	Suppliers
T 218 m-58 Forming Handsheets for Optical Tests of Pulp	Disintegrator, pump and press, press template, drying rings, blotting paper, Buchner funnel (porcelain), suction flask, filter paper, graduated cylinders	TMI
T 219 m-54 Bleach Requirement of Pulp	Jars, stirrer, water bath, accessories	TMI
T 221 m-51	Disintegrator, sheet machine, stirrer, stop watch, container, dipper, graduated cylinder	TMI
T 224 sm-45 Laboratory Processing of Pulp (Ball-Mill Method)	Disintegrator, ball mill	TMI
T 226 sm-52 Specific External Surface of Pulp	Reaction apparatus, hot plates, crucibles, porcelain casserole, stopwatch or stopclock, dissecting needle	TMI
T 227 m-50 Freeness of Pulp	Freeness tester, graduated cylinder, disintegrator, suitable bracket and dipper	TMI
T 231 sm-58 Zero-span Breaking Length of Pulp	Tensile tester, zero-span jaw attachment	TMI
T 231 sm-58 Zero-span Breaking Length of Pulp	Zero span grips	TAI
T 232 sm-58 Fiber Length of Pulp by Projection	Fiber projector	TAI
T 233 sm-53 Fiber Length of Pulp by Classification	Fiber classifier, hot plate, steam heated cylinder, or temperature-regulated drying oven, balance, projector, disintegrator, accessories	TMI
	Fiber classifier	BB TAI
T 234 sm-58 Coarseness of Pulp Fibers by Projection	Fiber projector	TAI
T 403 m-53 Bursting Strength of Paper	Mullen tester	TMI BFP
T 404 m-50 Tensile Breaking Strength of Paper and Paperboard	Tensile tester	TMI TAI
T 410 m-45 Basis Weight of Paper and Paperboard	Basis weight scale	TMI TAI
T 411 m-44 Thickness and Density of Paper	Micrometer	TMI
T 413 m-54 Ash in Paper	Weighing container, thermometer, drying oven, chemical balance, desiccator	TMI
T 414 m-49 Internal Tearing Resistance of Paper	Tearing tester	TMI TAI
T 423 m-50 Folding Endurance of Paper	Schopper-type folding endurance tester, MIT-type folding endurance tester	TMI
T 426 m-46 Bulking Thickness of Paper and Paperboard	Micrometer	TMI
T 429 m-48 Alpha, Beta, and Gamma-Cellulose in Paper	Disintegrator, Koerner type	TMI
T 430 m-52 Copper Number of Paper and Paperboard	Grinder, Koerner type	TMI
T 433 m-44 Water Resistance of Paper and Paperboard (Dry Indicator Method)	Penescope and dry indicator powder	TAI
T 435 m-52 Hydrogen Ion Concentration (pH) of Paper Extracts	pH Meter, disintegrator, Koerner type	TMI
T 441 m-58 Water Absorptiveness of Nonbibulous Paper Paperboard (Cobb Test)	Specimen holder, stopwatch	TMI
T 446 m-48 Moldability (Crease Retention) of Wrapping Paper	Protractor, weights, method B weights, method A, flat surface, stop watch	TMI
T 447 m-53 Moisture Expansivity of Paper	Expansivity tester	TAI
T 448 m-49 Water Vapor Permeability of Paper and Paperboard	Test dishes, template, Analytical balance	TMI
	Test dishes	TAI
T 450 m-44 Pentosans in Paper	Disintegrator	TMI
T 451 m-45 Rigidity, Stiffness and Softness of Paper and Paperboard	Softness-stiffness tester, rule graduated in millimeters, scale for basis weight, micrometer	TMI
		TAI
T 453 m-48 Heat Test for Relative Stability of Paper	Oven, folding endurance tester	TMI
T 456 m-49 Wet Tensile Breaking Strength of Paper and Paperboard	Tensile tester, Finch wet strength attachment	TMI TAI
T 457 m-46 Stretch of Paper and Paperboard	Tensile tester, clamp, loading mechanisms, indicating device	TMI TAI TMI
T 460 m-49 Air Resistance of Paper	Air resistance tester	
T 462 m-43 Printing Ink Permeation of Paper	Printing ink permeability tester	TMI
T 463 m-52 Adhesiveness of Gummed Paper Tape	Gummed tape tester	TMI
T 463 m-52 Adhesiveness of Gummed Paper Tape	Gummed tape tester	TAI
T 464 m-45 Water Vapor Permeability of Sheet Materials at High Temperature and Humidity	Test dishes	TMI TAI
T 466 m-52 Degree of Curl and Sizing of Paper	Curl and size tester, stopwatch	TMI TAI
T 469 sm-55 Flexural Resistance and Deflection of Fiberboard Bookbinding	Tensile tester with scale, flexure loading device	TMI TAI
T 470 m-54 Edge Tearing Resistance of Paper (Finch Method)	Finch edge-tear stirrup, tensile tester	TMI TAI
T 472 m-51 Compression Resistance of Paperboard (Ring Crush Test)	Compression machine, specimen holder, cutting device	TMI
T 476 m-51 Abrasion Loss of Paper and Paperboard	Abrading instrument, resurfacing turntable, brush	TMI
T 478 sm-52 Ink-Erasing Quality of Paper	Abrading machine and accessories, micrometer	TMI

Method	Apparatus	Suppliers
T 479 sm-48 Smoothness of Printing Paper	Bekk smoothness tester, Gurley-Hill S-P-S smoothness tester	TMI
T 480 m-51 Specular Gloss of Paper at 75°	Gloss Meter—75°	TMI
T 481 sm-52 Fiber Orientation in Paper (Zero-span Tensile Strength)	Tensile tester, zero-span jaws	TMI TAI
T 482 m-52 Water Vapor Permeability of Sheet Materials at 0°F.	Test dishes	TAI
T 637 sm-54 Viscosity of Starch and Starch Products	Corn Industries Research Foundation viscometer	GSC
T 643 sm-54 Dextrenizing Value of Enzymes	Hellige alpha-amylase color disk no. 620-S5 used in conjunction with a no. 605 Hellige	HI
T 644 m-58 Tensile Strength of Paraffin Wax	Tensile strength tester	TAI
T 648 sm-54 Viscosity of Coating Clay Slurry	Viscometer, mixer	TMI
T 652 sm-57 Blocking Range of Paraffin Wax	S.O.D. blocking point tester	TAI
T 800 m-50 Drum Test for Fiberboard Shipping Containers	Revolving drum, 7 ft.	TMI
T 801 sm-44 Impact Resistance of Fiberboard Shipping Containers	Incline impact testing device	TMI
T 803 m-50 Puncture Test of Container Board	Puncture tester, complete	TMI
T 804 m-45 Compression Test of Fiberboard Shipping Containers	Compression testing machine	TMI
T 806 sm-46 Adhesiveness of Seals and Closures for Packages	Tensile testing machine, shallow tray or dish, blotting paper	TMI
T 1001 sm-51 Forming Structural-Type Boards for Physical Tests of Pulp	Board machine, stirrer, screens, press, plates, board drier, scale	TMI
T 1001 sm-51 Forming Structural-Type Boards for Physical Tests of Pulp	Williams test board former	WAC
T 1002 sm-51 Standard Drainage Time of Pulp for Insulating Board	Disintegrator, container, container-stirrer, drainage tester, drainage tester stirrer, accessories	TMI
T 1002 sm-51 Standard Drainage Time of Pulp for Insulating Board	Drainage tester	WAC
T 1003 sm-55 Flexural Resistance and Deflection of Insulating Fiberboard	Testing machine, loading head and supports, scale	TMI
T 1003 sm-55 Flexural Resistance and Deflection of Insulating Fiberboard	Flexural rigidity fixture for tensile tester	TAI

Key to Abbreviations

AEC	Abbe Engineering Co., 420 Lexington Ave., New York 17, N. Y.
BB	The Bauer Bros. Co., Burt & Sheridan, Springfield, Ohio
BFP	B. F. Perkins & Son, Inc., Holyoke, Mass.
EHS	E. H. Sargent & Co., 4647 W. Foster Ave., Chicago, Ill.
GSC	The Gaertner Scientific Corp., 1201 W. Wrightwood Ave., Chicago 14, Ill.
HI	Hellige, Inc., 877 Stewart Ave., Garden City, N. Y.
HMC	The Hermann Manufacturing Co., 211 North Columbus St., Lancaster, Ohio
TAI	Thwing-Albert Instrument Co., Penn St. & Pulaski Ave., Philadelphia 44, Pa.
TMI	Testing Machines, Inc., 72 Jericho Turnpike, Mineola L. I., N. Y.
VIW	Valley Iron Works Co., Appleton, Wisc.
WAC	Williams Apparatus Co., Inc., 12 Herald Bldg., Watertown, N. Y.
WC	Will Corporation, Rochester, N. Y.



TAPPI PROFILES

The biographical sketch on this page is the first of a series which will appear in this magazine under the heading "TAPPI Profiles."

These features will present to the readers of Tappi, *leading members of the Association. It is hoped that by describing their careers and, in particular, their activities in TAPPI, these outstanding members will become more widely known and at the same time will help to reveal further the highly professional character and personality of the Association.*

Because James R. Lientz is an outstanding member of the Association as well as its President, he has been selected for the first TAPPI Profile.

James R. Lientz

James R. Lientz, 26th president of TAPPI, was born in Los Angeles, Calif., on July 28, 1913.

After completing his primary education at the Kansas City Junior College in Kansas City, Mo., he went to the University of Michigan, Ann Arbor, from which he graduated in 1937 with an M.S. degree in Chemical Engineering. Upon graduating, Mr. Lientz went to work for Swenson Evaporator, Harvey, Ill., and it was there that he first came in contact with the pulp and paper industry. In his seven years with Swenson Evaporator he served as sales representative and later as manager, Pulp and Paper Division.

He joined Union Bag and Paper Corp., Savannah, Ga. (now Union Bag-Camp Paper Corp.) in 1940 as assistant chief chemist, and later became chief chemist. In 1946, after a three and a half-year absence from Union Bag, Mr. Lientz rejoined the corporation as superintendent of the mill technical department, later to become general superintendent and manager of the Pulp and Paper Division. In 1955 he was promoted to manager of the Savannah Plant Union Bag-Camp Paper Corp., and in 1956 was named resident manager and vice-president of the corporation, in charge of production. He is presently vice-president—production, in charge of the Savannah plant, the world's largest pulp and paper mill, and of the corporation's converting operations. In addition to being responsible for all operations in Savannah, he is also in charge of the operations of: box plants in Chicago, Ill., Trenton, N. J., Jamestown, N. C., Spartanburg, S. C., and Lakeland, Fla.; bag factories in Richmond, Va., New Hope, Pa., and St. Louis, Mo.; and a honeycomb plant in Glens Falls, N. Y.

The current president of TAPPI has been a member since 1940. In 1950 he became chairman of the Chemical Engineering Committee, which under his chairmanship undertook an intensive study of corrosion in alkaline digesters. To carry out the important details of the investigations, he helped organize and was made chairman of the Digester Corrosion Subcommittee. Of the many publications which resulted from the valuable work of the subcommittee perhaps the most widely known are the five-year report entitled "Five-Year Investigation of Digester Corrosion by the TAPPI Chemical Engineering Subcommittee," published in *Tappi* 38, No. 6, 373 (1955), and TAPPI Monograph No. 12, "Inspection of Digesters." This Monograph finally gave to the paper industry a long-awaited manual for the routine maintenance and safety control of digesters.

As a member of the TAPPI Executive Committee since 1952, Mr. Lientz has served on many committees of the executive branch. In 1954 he was the Executive Committee Representative for the Southeastern Section, and a member of both the Local Sections and the Publications Management Committee; he became chairman of the former committee in 1955 and of the latter two years later. Also in 1957, he became chairman of the Awards Committee and a member of four other committees: Finance, Research Appropriation, Retirement and the American Paper and Pulp Association (APPA) Technical Committee. He is still a member of the last three committees and represents TAPPI on the APPA Technical Committee. Since 1958 he has been a member of the Investment Advisory Committee.

In February, 1959, at the 44th Annual Convention, he was elected president of TAPPI.

Despite his professional and TAPPI activities and the

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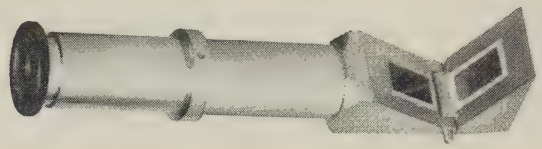

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immediate demands made on a father of three children, James Lientz' encompassing interests enable him to shoulder the burden of service in many civic organizations. He is an elder of the First Presbyterian Church of Savannah; a member of the Advisory Committee, Citizens and Southern National Bank; a board member on the Savannah District Authority; director of the Savannah Symphony Society, Inc., The Savannah Foundation, Inc., and the Savannah Area Solicitations Review Board; a member of the board of Trustees of the Georgia Infirmary and of the Warren A. Candler Hospital; on the board of managers of the Union Society of Savannah (Bethesda Home for Boys); a vice-president of the United Community Services, and chairman of the Campaign Cabinet; chairman of the 1959 United Community Appeal; and a member and former chairman of the Committee on Services to Handicapped Children.

His concern for the welfare of youth is further expressed by the interest he takes in the Boy Scouts of America. Once president of the Coastal Empire Council, B.S.A., he is now member-at-large of the National Council, member of the Order of the Arrow, and holder of the Silver Beaver Award, B.S.A.

He also belongs to The Savannah Rotary Club, Savannah Golf Club, Oglethorpe Club, A.I.Ch.E., and American Pulpwood Association.

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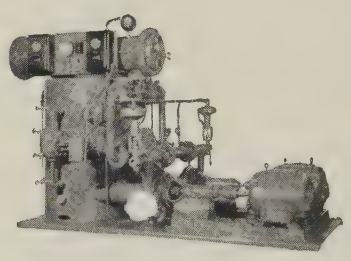

The Stickle Vacuum Pump is made exclusively for paper mills. It's easy to service. All floats housed in exterior float boxes. Interchangeable, removable vacuum chamber is above tank. Inspection of ejector tubes may be made without moving motor, pump or piping. Uses standard horizontal motor and pump. For vacuums to 28". Built-in heat exchanger eliminates costly cooling equipment. Recirculation of hurling water condenses flash vapors and releases air and gases to atmosphere. No close clearances to cause wear. Performance guaranteed. Single and duplex models. Write for free literature.

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TAPPI NOTES

Eleven Years of Tappi

ELEVEN years ago this month *Tappi* was born. By today's standards the first issue was a modest 116-page publication, but in its short life *Tappi* has grown steadily, both in size and in stature, as a professional journal.

W. F. Gillespie, president of TAPPI in 1949, wrote in the first issue, "The quality of the literature on the many phases of pulp and paper technology can be improved, and it shall be the main objective of *Tappi* to accomplish this. *Tappi* will be a balanced presentation of the science of our industry. It will attempt to appeal to our membership as a whole but always deal with facts. Quality is our watchword."

Quality has continued to be the watchword. *Tappi's* reputation today is a tribute to the authors, to the editorial boards, and especially to R. G. Macdonald and R. T. Bingham, who have zealously maintained the standards of excellence established in the first issue.

The last eleven years have brought many changes to *Tappi* and to the Association. The magazine has expanded to an average monthly issue of over 280 pages. The Technical Sections have doubled in size. The dynamic growth in TAPPI membership from under 4000 in 1949 to almost 10,000 today has produced a corresponding surge in magazine space devoted to Association activities. There has been a gratifying increase in advertising. New features such as "Industry Notes," "Employment Service," and "Book Reviews" have been added to enhance the value of the magazine to its readers.

Nevertheless, the growth of the magazine and the Association has also introduced some difficulties. Unfortunately, few readers have the time to read carefully and digest a monthly journal of *Tappi*'s size. For many readers *Tappi* must be skimmed hurriedly and set aside hopefully for an hour of leisurely reading which may never materialize.

Partly because of limited reading time and partly because of the Association's continuing growth and broadening interests, it has become increasingly difficult for individual members to keep informed of Association activities and policies. Many members today have only a vague concept of TAPPI objectives and almost no knowledge of the TAPPI programs aimed toward achieving these objectives. The Association leaders, who have been so instrumental in the success of these programs, are known only by name to large numbers of TAPPI members.

In an effort to overcome these difficulties the editorial staff is introducing some changes in the layout and contents of this and subsequent issues of *Tappi*. Basically these innovations are aimed at improving the readability of the magazine and at acquainting the readers with Association programs and policies.

The "Table of Contents" has been expanded from one to three pages. This will permit the deserved use of a full page for the technical section papers, the heart of *Tappi* as a professional journal. Equally important, it will provide space for considerable detail in the portion devoted to Association activities. Thus the reader will be able to locate quickly the reports of interest to him without having to search for them. A calendar of coming events will be included for ready reference, and the use of a larger type size will facilitate reading.

As a further aid the reader will find increased use of mastheads, subheadings, and boldface type in the general section of the magazine.

A new feature entitled "TAPPI Profiles" will be a monthly biographical sketch of an outstanding TAPPI member written by the editorial staff. It is hoped that this page will better acquaint readers with their TAPPI leaders and also with the Association programs in which these men have played significant roles. J. R. Lientz, president of TAPPI, has been selected for the first of this series.

Another innovation is *this* page, "TAPPI Notes." Formerly this name referred to the section of "Personal Mention" listing new assignments for TAPPI members. Henceforth "TAPPI Notes" will identify *this* page of editorial comment on Association activities and developments. This will enable the officers and headquarters staff to bring Association matters of general interest to the attention of the membership.

Over the eleven years of its short life *Tappi* has represented one of the Association's greatest services to its members and to the industry. We hope that the changes initiated in this issue will further augment *Tappi*'s value as a professional journal and as the official publication of our Association.

PHILIP E. NETHERCUT

PERSONAL MENTION

New TAPPI Members

Millicent W. E. Allen, Librarian, Imperial Tobacco Co. of Canada Ltd., Montreal, Que., Canada.

Maurice R. Asselin, Research Engineer, St. Regis Paper Co., Carthage, N.Y., a 1959 graduate of University of New Hampshire.

Gloria A. Bernier, Research Mathematician, St. Regis Paper Co., Carthage, N.Y., a 1945 graduate of St. Lawrence University.

Robert B. Boies, Project Engineer, Cincinnati Industries Corp., Cincinnati, Ohio, a 1947 graduate of University of Kentucky.

George G. Clark, Assistant Technical Director, The Mead Corporation, Kingsport, Tenn., a 1951 graduate of Georgia Institute of Technology.

Amando Clemente, President, Aclem Paper Mills, Inc. and University Paper Mills, Diliman, Rizal, P.I., a 1920 graduate of the University of Chicago with a Ph.D. degree.

Harry E. Earl, Plant Manager, Finch, Pruyn & Co., Glens Falls, N.Y.

Tage L. Elers, Graduate Student, College of Forestry, University of Washington, Seattle, Wash., a 1959 graduate of University of Washington.

Per Engstrom, Technical Sales, Holmens Bruks- och Fabriks AB, Norrkoping, Sweden, a 1959 graduate of Royal Institute of Technology.

Jack P. Feldstein, Sales Representative, Champion Paper Export Corp., San Juan, Puerto Rico, Attended New York University.

William S. Gorman, Jr., Head, Research Laboratory, Hallmark Cards, Inc., Kansas City, Mo., a 1950 graduate of Purdue University.

Alan C. Hamilton, Research Chemist, Oxford Paper Co., Rumford, Me., a 1957 graduate of University of Maine.

Haruo Hashimoto, Laboratory Worker, Gooh Chemical Industry Co., Ltd., Kyoto, Japan, a 1958 graduate of Kyoto University.

Leonard F. Hayne, Application Engineer, Allis-Chalmers Mfg. Co., Milwaukee, Wis., a 1949 graduate of Marquette University.

Robert O. Koch, Chemist, Gaylord Container Div., Crown Zellerbach Corp., Bogalusa, La., a 1956 graduate of Millsaps College.

Victor M. Levey, Technical Director, Tragacanth Importing Corp., New York, N.Y., a 1958 graduate of Brooklyn College.

James C. Lindsey, Research Chemist, Orr Industries Div., Ampex Corp., Opelika, Ala. Attended Pennsylvania State College.

Edmund J. McLohne, Marketing Manager, UNI-GUM Div., T. M. Duche & Sons, Inc., New York, N.Y., a 1950 graduate of University of Maine.

Kiyoshi Numabe, Chief Engineer, Rengo Shiki Co., Ltd., Osaka, Japan, a 1931 graduate of Keio University.

Harry N. Packard, Graduate Research Assistant, University of Maine, Orono, Me., a 1959 graduate of University of Maine.

Walter G. Peck, Editorial Representative, Paper Mill News, New York, N.Y.

Emanoel Peixoto, Technical Superintendent, Companhia Mineira de Papeis, Cataguases, Brazil.

Bastiaan H. Pot, Managing Director, N.V. Noord-Nederlandsche Machinefabriek, Winschoten, Holland, a 1942 graduate of Technical University, Zurich, Switzerland.

William S. Shires, Service Engineer, Albany Felt Co., Albany, N.Y., a 1950 graduate of Lowell Technological Institute.

Charles T. Smith, Assistant Marketing Manager, The Babcock & Wilcox Co., Barberton, Ohio. Attended Rensselaer Polytechnical Institute.

Marvin H. Smith, Jr., Director of Research, Southern Sizing Co., East Point, Ga., a 1949 graduate of Georgia Institute of Technology.

Raul Szuchman, General Manager, Papelera Interamericana S.R.L., Buenos Aires.

Robert L. Thomsen, Salesman, American Marietta Co., Seattle, Wash., a 1950 graduate of University of Washington.

James J. Turek, Jr., Technical Representative, The Hubinger Co., Keokuk, Ia.

George J. Wagner, Assistant Paper Mill Superintendent, Marathon, Div. of American Can Co., Green Bay, Wis., a 1950 graduate of University of Wisconsin.

William F. Waldeck, Consultant, C. K. Williams & Co., Easton, Pa., a 1933 graduate of New York University with a Ph.D. degree.

Eduard F. Weiss, Superintendent, Photographic Div., Hugo Albert Schoeller, Dueren, Germany, a 1924 graduate of Technical University, Dresden.

Dennis Wilkinson, Works Director, Backhouse & Coppock Ltd., Cheshire, England, Attended Macclesfield College of Technology.

Julius V. Wolff, Sales Representative, Nopco Chemical Co., Newark, N.J., a 1939 graduate of Syracuse University.

Roy B. Young, Jr., Chief Engineer, The Moore & White Co., Philadelphia, Pa., a 1951 graduate of Lehigh University.

Appointments

Frederick A. Adams has been transferred from Valdosta, Ga., to Jacksonville, Fla., as Assistant Mill Manager of Owens-Illinois, Mill Division.

Kenneth Arnold is now Vice-President for Stein, Hall & Co., Inc., New York, N. Y.

Peter A. Absalom, formerly with Bradshaw's Ltd., is now Technical Manager for Milprint, Inc., Downingtown, Pa.

Leslie K. Bickell has been transferred from Seattle, Wash., to Sitka, Alaska, as Technical Director of Alaska Lumber & Pulp Co.

Francis O. Boylan is now Manager for Manufacturing for Crown Zellerbach Corp., San Francisco, Calif.

Benton R. Cancell has been transferred from Rhinelander, Wis., to New York, N. Y., as Executive Vice-President of St. Regis Paper Co.

Thomas Capstick, Jr., formerly New York Sales Manager for Dixon Chemical & Research, Inc., is now General Manager for Dixon International, Inc., Bloomfield, N. J.

Sidney M. Collier is now Vice-President & General Manager for Spaulding Pulp & Paper Co., Newberg, Ore.

Howard W. Comey, formerly Sales Technical Representative for the Borden Co., is now with New Product Development, Borden Chemical Co., Leominster, Mass.

Rene V. Cormier, Student of Lowell Technological Institute, is now Graduate Student at St. Louis University, St. Louis, Mo.

William Deskins, formerly Consulting Engineer, is now Project Engineer for Hudson Pulp & Paper Corp., Southern Division, Palatka, Fla.

Richard H. Dillinger, formerly with Paisley Products, Inc., is now Assistant Manager, Paper Division for Morningstar-Paisley, Inc., New York, N. Y.

Dan D. Downs is now Sales Manager, Distillation Products Div., Capital City Products Co., Columbus, Ohio.

Paul E. Grunder has been transferred from New York, N. Y., to Englewood Cliffs, N. J., as Sales Engineer for Dow Corning Co.

Arne Hallencreutz, formerly with American SF Products, Inc., is now Vice-President for AB Svenska Flaktfabriken, Stockholm, Sweden.

William P. Hamill is now Special Treatments Chemist, Product Dept., Paper & Board Divisional Laboratory for Albert E. Reed & Co. Ltd., Larkfield, nr., Maidstone, Kent, England.

John A. Harpham, formerly Research Chemist with Hercules Powder Co., is now Research Supervisor for Hercules Research Center, Wilmington, Del.

Robert G. Hitchings is now Associate Professor for North Carolina State College, School of Forestry, Raleigh, N. C.

Gaston L. Hollimon, formerly with Longview Fibre Co., is now Pulp Mill Superintendent for Tennessee River Pulp & Paper Co., Counce, Tenn.

Bruce I. Howe, formerly with Canadian Pulp & Paper Mills Ltd., is now Trainee for Quebec North Shore Paper Co., Baie Comeau, Que., Canada.

James D. Johnson, formerly with Container Corp. of America, is now Assistant Plant Manager for Continental Can Co., Piermont, N. Y.

Daniel E. Kane, formerly with Fibreboard Products, Inc., is now Group Leader, Paper & Fibre Section, Research & Development Laboratory for National Vulcanized Fibre Co., Yorklin, Del.

John T. Kiley, formerly with James Fleet Organization, is now President, Moore Waste Paper Co., Inc., Elmhurst, Ill.

Lloyd T. Krumm, is now Manager of Bleached Board Sales for International Paper Co., New York, N. Y.

Harold M. Lacey, formerly with Greater Winnipeg Sanitary District, is now with Technical Sales for Buckman Laboratories (Canada) Ltd., Montreal, P. Q.

Charles G. Lavender, formerly with Buckeye Cellulose Corp., is now with Bulk Sales for Procter & Gamble Distributing Co., Cincinnati, Ohio.

George R. Lawson is now General Manager, Chemical Specialties Div., Pennsalt Chemicals Corp., Philadelphia, Pa.

Daniel V. Lent, formerly with Alton Box Board Co., is now Technical Salesman for Morningstar-Paisley, Inc., Jacksonville, Fla.

John R. Lindquist is now Manager Paper & Corrugated Dept., Stein Hall & Co., Inc., New York, N. Y.

William F. Llewellyn, formerly with Marathon Div., American Can Co., is now Senior Project Engineer for Marathon Southern Corp., Naheola, Ala.

Manfred Luthgens, formerly of West Virginia Pulp & Paper Co., is now Group Leader, Paper Chemicals Lab., Nalco Chemical Co., Chicago, Ill.

Ernest C. Manders, formerly with Kimberly-Clark Corp., is now Mill Manager for Menasha Wooden Ware Corp., Menasha, Wis.

Robert D. McPheters, formerly with Gould Paper Co., is now Research Associate for Fiber Products Research Center, Inc., Beaver Falls, N. Y.

Byron C. Miller is now Production Assistant to General Superintendent, P. H. Glatfelter Co., Spring Grove, Pa.

Edward A. Miller, formerly with Mead Containers, Inc., is now Production Manager for Crown Corrugated Containers Corp., Greensburg, Pa.

Rudolf G. Minarik is now Consulting Engineer for Kimberly-Clark Corp., Neenah, Wis.

Wayne J. Mitchell is now District Manager for Link-Belt Co., Portland, Ore.

Michael J. Moriarty, formerly with Cupples-Hesse Corp., is now Development Chemist for Standard Packaging Corp., Johnston Foil Div., St. Louis, Mo.

W. D. Mosher, has been transferred from Three Rivers, Que., to Grand'Mere, Que., Canada, as Assistant Manager of Manufacturing of Consolidated Paper Corp. Ltd., Head Office.

Michael D. Mullin is now Regional Vice-President for Corn Products Sales Co., New York, N. Y.

Malcolm J. Odell, formerly with Ludlow Papers, Inc., is now Product Development Manager for Crocker, Burbank Papers, Inc., Fitchburg, Mass.

David E. Peakes has been transferred from Cloquet, Minn., to Brainerd, Minn., as Chemical Engineer, Technical Service Dept. of Northwest Paper Co.

David W. Peat is now Manager of Manufacturing for Gilman Paper Co., Gilman, Vt.

Henry B. Pratt, formerly with Stevenson & Rubens, is now a Partner of Rubens & Pratt, Seattle, Wash.

David L. Rawls is now Vice-President in charge of Sales for Dixon Chemical & Research, Inc., Bloomfield, N. J.

James W. Replogle, Jr., formerly with Rayonier, Inc., is now Shift Superintendent for Pannellit Service Corp., Skokie, Ill.

Robert C. Ridings has been transferred from Philadelphia, Pa., to Chicago, Ill., as Plant Engineer of Container Corp. of America.

Arthur L. Ross is now Vice-President and Assistant General Manager for International Paper Co., Mobile, Ala.

Richard C. Savage, formerly with the U. S. Army Chemical Corp., is now Chemical Engineer, Pandia Div., The Black-Clawson Co., Hamilton, Ohio.

William H. Schlafge has been transferred from International Falls, Minn., to Minneapolis, Minn., as Director of Research, Pulp & Paper, Minnesota & Ontario Paper Co.

Lutz Gerhart Schneider, formerly student at the Technical University of Darmstadt, Germany, is now Assistant to Technical Director for Gebr. Buhl K. G., Ettlingen/Baden, West Germany.

Clark E. Snook is now Regional Manager, Mid-Atlantic Area for Nopco Chemical Co., Newark, N. J.

Joseph N. Steiner, formerly with John W. Bolton & Son, is now Sales Engineer for E. D. Jones Corp., Pittsfield, Mass.

Ernest G. Steinhauer, formerly with West Virginia Pulp & Paper Co., is now Staff Engineer for Beloit Iron Works, Beloit, Wis.

Victor J. Sutton is now Vice-President & General Manager for Bowaters Southern Paper Corp., Calhoun, Tenn.

Hans Unander-Scharin, formerly of A & P Corrugated Box Corp., is now Production Manager, Merrimac Paper Co., Inc., Lawrence, Mass.

Ernst A. Wendt, formerly with Marathon Corp., is now Plant Manager, Stonebridge Paper Div., Personal Products Corp., Wilmington, Del.

Fritz M. K. Werdouschegg, formerly with Case Brothers, Inc., is now Project Leader, Product Dev. Dept., Hudson Pulp & Paper Corp., Palatka, Fla.

Robert C. Witt has been transferred from Chicago, Ill., to Midland, Mich., as Product Manager, Sales, for Dow Chemical Co.

Kenneth E. Youngchild has been transferred from Mobile, Ala., to New York, N. Y., as Sales Manager, Paper Chemicals Dept., for American Cyanamid Co.

INDUSTRY NOTES

North American

Statistics

*Paper and Paperboard**

Production of paper and paperboard in September, 1959, totaled an estimated 2,850,000 tons, bringing the 9-month estimate for this year to 25.4 million tons, which represents an increase of 12.4% above the comparable 1958 period. Paper production for the first three quarters of 1959 was 11.1 million tons, an increase of 11.3% above 1958; paperboard production was 11.8 million tons, an increase of 13.0% and construction paper and board production was 2.5 million tons, 14.4% above a year ago.

Paperboard

Total paperboard production alone during the first 10 months of 1959 reached 13,350,000 tons. This 10-month production is higher than the annual production in 5 years of the past decade—1950 to 1954. In 8 of the 10 months of 1959 average weekly production of paperboard has exceeded 300,000 tons.

Paperboard demand will climb in 1960 to 16.2 million tons, 2% above 1959's high level, P. W. Hoguet, president of the Econometric Institute told paperboard officials at the 27th Annual Meeting of the National Paperboard Association.

It is estimated that by 1965 paperboard demand in the United States would reach 20.1 million tons, 26% above estimated production this year, and by 1975 paperboard demand will total 28.1 million tons annually, 77% above current levels.

*Newsprint**

Production of newsprint in North America during October, 1959, amounted to 770,506 tons—an all-time high for any month on record—and was 10.2% above the October, 1958, volume. Shipments totaled 782,030 tons in October, 1959, and were 9.5% greater than shipments in the corresponding month of 1958. Output in the United States of 177,325 tons represented a historical monthly peak while shipments totaled 179,472 tons.

Canadian production of 593,181 tons exceeded that of any previous month while shipments aggregated 602,558 tons. Output in the United States was up from October, 1958, by 14.6% while that in Canada showed an increase of 9.0% over the volume of a year ago. Shipments from United States mills in October, 1959, as compared to October, 1958, rose by 13.1% while mills in Canada shipped 8.5% more.

North American production in the first 10 months of 1959 amounted to 6,920,347 tons and was 358,594 tons or 5.5% greater than in the comparable period of 1958. Cumulative output of United States mills at the end of October, 1959, totaled 1,627,948 tons—an all-time high for this period—and was 167,416 tons or 11.5% more than the volume produced a year earlier. Canadian production amounted to 5,292,399 tons and was 191,178 tons or 3.7% greater than in the first 10 months of 1958.

North American manufacturers' stocks on Oct. 31, 1959, aggregated 217,336 tons compared with 228,860 tons on the last day of the preceding month and 230,933 tons at the end of October, 1958. The continental total was composed of 19,184 tons and 198,152 tons held by United States and Canadian mills, respectively, and inventories were higher than a year ago by 550 tons in the former instance and lower by 14,147 tons in the latter.

Imports of newsprint paper from overseas during the month of September, 1959, were 3925 tons greater than in the corresponding month of 1958. Receipts from Canada also were greater by 66,575 tons, thus total imports exceeded those of a year ago by 70,500 tons or 18.0%.

Imports from overseas during the first 9 months of 1959 were 21,999 tons above the volume thereof in the comparable period of 1958. The volume of receipts from Canada also was greater than in 1958 by 204,022 tons, thus the increase in imports from all sources amounted to 226,021 tons or 6.1%

In the first 9 months of 1959 tonnage of an external origin constituted 73.6% of the indicated new supply of newsprint in the United States as against 74.6% a year ago.

Pulpwood

Total receipts of pulpwood for the first 8 months of 1959 were 24,805,000 cords, an increase of 2,638,000 cords over receipts for the comparable period of 1958. Domestic receipts were 2,741,000 cords above the year earlier total, while imports declined 103,000 cords. All of the regions showed increases ranging from 3% in the Northeast, to 15% in the South Atlantic.

Pulpwood consumption in the first 8 months of 1959 was 25,749,000 cords, an increase of 2,911,000 cords over the same period of 1958. All of the regions showed increases ranging from 1% Northeast to 18% South Atlantic.

Inventories of pulpwood at the end of August were 4,995,000 cords, an increase of 80,000 cords above July, 1959, and a 1,066,000 cords decline from August last year. Four of the regions showed decreases ranging from 15% North Central to 25% West. The South Central region showed an increase of 4%.

Wastepaper and Other Fibrous Materials

August wastepaper receipts were 767,000 tons; for the first 8 months of this year they totaled 6,203,000 tons, an increase of 555,000 tons above receipts for the first 8 months of last year.

Wastepaper consumption for August was 796,000 tons; the January–August total was 6,110,000 tons, an increase of 422,000 tons above consumption for the first 8 months in 1958.

Inventories at the end of August were 514,000 tons, a decrease of 29,000 tons below July, and 68,000 tons above inventories at the end of August, 1958.

Consumption of total fibrous materials for August was 879,000 tons; the January–August total was 6,812,000 tons, an increase of 1,135,000 tons above consumption for the first 8 months of 1958.

Inventories of total fibrous materials at the end of August were 876,000 tons, 48,000 tons below July, and 61,000 tons above inventories at the end of August, 1958.

* APPA, *Monthly Statistical Summary* 37, No. 10 (1959).
* Newsprint Service Bureau, *Bulletin* No. 502 (1959).

Wood Pulp

Production of wood pulp for the first 8 months of 1959 was 16,089,000 tons, an increase of 2,149,000 tons above the comparable 1958 total.

Wood pulp consumption in the manufacture of paper and board for the first 8 months of 1959 was 16,668,000 tons, an increase of 2,232,000 tons above consumption for the first 8 months of 1958.

Imports of wood pulp for January–August 1959, were 1,615,440 tons, an increase of 30% above imports for the first 8 months of last year. Imports from Canada for the first 8 months of 1959 were 1,322,481 tons, an increase of 14% over the comparable period of 1958. Overseas imports for the first 8 months of 1959 were 293,029 tons as compared to 156,958 tons in the first 8 months of 1958.

Exports of wood pulp for the first 8 months of this year were 403,822 tons, as against 338,144 tons exported during the same period last year.

Inventories of wood pulp at paper and board mills at the end of August were 543,000 tons a decrease of 4000 tons below inventories at the end of July and 6000 tons below inventories at the end of August, 1958.

Overseas

Western Europe's Pulp and Paper Industry

A recent report by the Organization for European Economic Cooperation (The Pulp and Paper Industry in Europe—Developments between 1950 and 1957; The Situation in 1958; Prospects for 1963–65), brings out the following salient facts: The 17-nation group, plus Finland, had a total of 2000 pulp, paper, and board mills in 1958, employing 420,000 workers, with a value of paper and board production amounting to $3.5 billion. Further data for 1958 were as follows: paper and board production, 16.2 million metric tons; pulp production, 11.13 million metric tons; consumption of fibrous raw materials, 16.1 million metric tons; and apparent consumption of paper and board, 15.3 million metric tons (70% increase from 1950 to 1958).

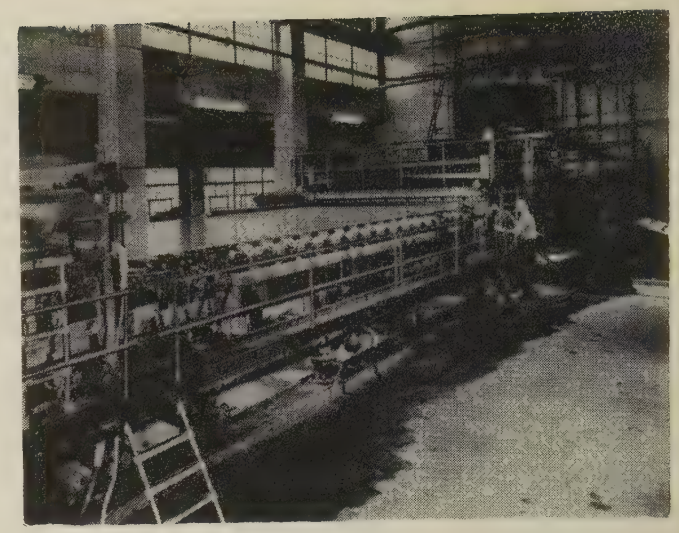
On paper and board consumption, the reports gives this information. National income of the OEEC countries rose 37%, i.e., by an average of 4.6% per year; apparent consumption and production of paper and board rose 66 and 57% respectively, i.e., by 7.5 and 6.6% per year on average. The increase of consumption was particularly marked for packaging paper and board (84% for kraft paper and board).

At a rate of increase of 4.5% in consumption of paper and board, it is expected that the demand for wood pulp from the OEEC countries and Finland should increase from 10.8 million metric tons in 1957 to about 13.8 million metric tons in 1963. By 1965, the paper industry in the OEEC countries and Finland is expected to consume 14.7 million metric tons of wood pulp, 6.1 million metric tons of wastepaper and 1.0 million metric tons of other materials, all to meet an anticipated paper and board production of 22 million metric tons. (Note: Copies of this detailed report are available for $1.50 from the OEEC Mission, 1346 Connecticut Ave., N. W. (Suite 1223), Washington 6, D. C.)

England

Black-Clawson Equips Townsend Hook Mill

The mill of C. Townsend Hook & Co., Ltd., Snodland, Kent, recently started up a new Black-Clawson paper machine designed to make magazine printing papers. The paper machine and the stock preparation system were supplied by Black-Clawson International, Ltd., London, which also engineered the flow of the new system and its instrumentation.



The new printing paper machine at C. Townsend Hook & Co. Ltd.

Stock Preparation System

The Snodland mill receives its pulp by barge. Overhead cranes unload the barges and carry the pulp to a storage yard, from which it is taken into the mill, as required, by overhead cranes and power trucks.

In the paper mill, the pulp bales are fed by specially designed conveyors to two-batch Hydrapulpers in which they are slushed at a high consistency. The two Hydrapulpers are installed so that they can be operated in two independent stock lines. At present, however, different types of pulps are not treated separately because the intermixing provides a desirable furnish for the class of paper now being made.

From the Hydrapulpers the pulp is pumped to the Hydrapulper dump chest, from which the flow of the system becomes continuous. Blended pulp is treated first in three 4B Hydrafiners and then goes to the machine chest where water is added by means of a consistency regulator. From the machine chest the stock receives final treatment in three Black-Clawson No. 2A jordans and is pumped through four Selectifier screens to a pressure controlled headbox of Black-Clawson design.

The instrumentation for this system consists of a simple master orifice meter with several slave orifice meters controlling the additives.

Paper Machine Details

The Black-Clawson headbox can operate under vacuum conditions for lower speed ranges or under pressure for higher speeds. The level in the headbox is kept consistent to insure that the evener rolls are always covered. This controls the flow of stock into and out of the box to best advantage.

The Black-Clawson paper machine is Hydroflyte design and produces a sheet of 168-in. trim. The headbox delivers stock through the cantilever fourdrinier which carries a wire approximately 185 in. wide × 90 ft. long.

The machine carries a suction couch, two suction presses, a smoothing press, and drying section with partially enclosed hood. The dry end is equipped with a Black-Clawson calender and a Black-Clawson Autoflyte reel of the horizontal type.

Argentina

Nuodex Products Co., a division of Heyden Newport Chemical Corp., announced that it has granted a license to Nuodex Argentina, S. A., to manufacture a complete line of paint driers and several specialty chemicals in the vicinity of Buenos Aires.

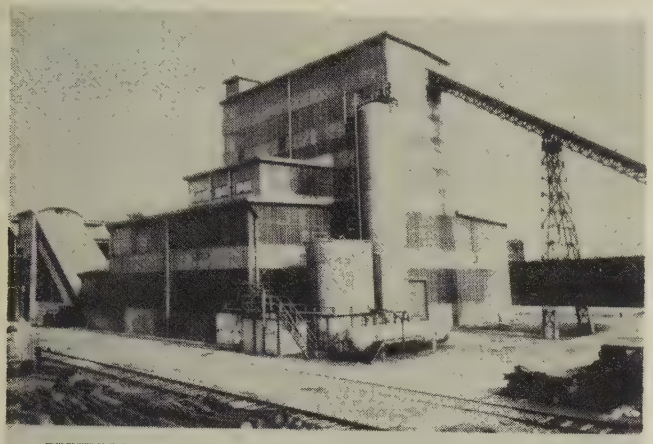
Nuodex Argentina was formed by Heyden Newport and a group of prominent English and Argentine businessmen,

headed by Cyril Taylor, president of Rodrap, S. A., of Buenos Aires.

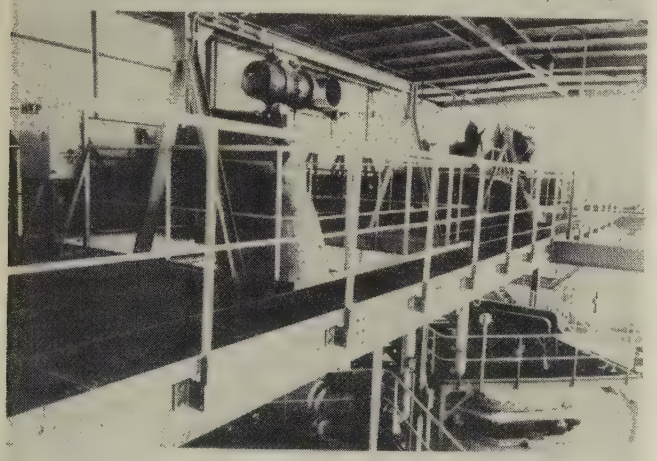
France

Chemi-Groundwood Pulp Mill Built

The French call it "pate chimico-mecanique." In English, it is called a chemi-groundwood plant. In either language, it describes the first plant of its kind built in Europe to meet the needs of European news-print manufacturers for pulp.



EXTERIOR OF EUROPE'S FIRST CHEMI-GROUNDWOOD PLANT BUILT AT CORBEHEM, FRANCE FOR SOCIETE F. BEGHIN, S. A.



INTERIOR VIEW OF CHEMI-GROUNDWOOD PLANT DESIGNED TO PRODUCE PULP FOR NEWSPRINT FROM EUROPEAN HARDWOOD.

A chemi-groundwood plant, designed to produce 100 tons of hardwood pulp a day, was built in the Pas de Calais area for the Societe F. Beghin, S. A., at Corbehem, France, by the Stone & Webster Engineering Corp.

French, British, and U. S. Engineers

The well-coordinated services of Stone & Webster Engineering Corp. and its affiliates were used effectively in the design and construction of this Beghin Plant. Process information was prepared in Boston. Engineering and drafting were directed by engineers from Boston and the London office of Stone & Webster Engineering Ltd., the British affiliate. Purchasing and inspection were done by engineers of the Paris office of Stone & Webster Engineering, S. A. Construction liaison services were provided by engineers from London and assistance on final commissioning by engineers from Boston.

Permits Use of Hardwoods

The Beghin chemi-groundwood mill, a valuable contribution to the European pulp and paper industry, provides an answer to the problem of utilizing economically the "hardwood" trees, from forests having an abundance of this raw material.

In most accessible areas, the "softwood" trees have been largely cut and sent to the paper mill because softwood is easier to process in the existing pulping plants. The hardwood trees have been left standing.

In these areas woodcutters had already installed expensive equipment for removing the softwoods, now largely depleted. The hardwoods are not only available but have re-seeded the cut areas and these new trees grow to papermaker's maturity in only 20 years. The Great Northern Paper Co. in the State of Maine faced and overcame very similar conditions in their forests.

Saving in Electric Power

The chemi-groundwood process used in the Beghin Plant in Corbehem, pressure cooks whole hardwood logs in a neutral sulfite solution, and after cooking they are ground in a conventional softwood grinder. The cooking loosens the lignin bond so that wood fibers are actually pulled off the log without appreciable damage together with the lignin surrounding the fibers so that the total bulk is still present. This softwood requires less grinding horsepower, thereby resulting in considerable savings in electric power and initial investment. Still another advantage of chemi-groundwood is its tear strength which is greater than regular groundwood, thereby making it possible to produce a satisfactory newsprint from a blended stock of groundwood, chemi-groundwood and a reduced percentage of sulfite pulp.

Soda ash, sulfur dioxide, and caustic soda are received by rail. Soda ash is stored as a monohydrate slurry, sulfur dioxide as a liquid and caustic soda as a 50% solution.

Sulfite solution is made in batches by reacting measured amounts of aqueous soda ash solution with a measured amount of sulfur dioxide. The resultant sulfite solution is stored and used to fortify the cooking liquor after each cook.

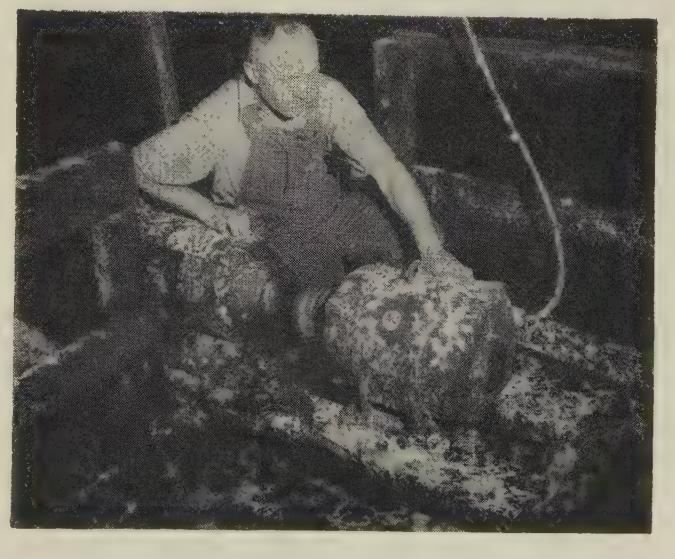
Paper Manufacturers and Converters

C. H. Dexter & Sons, Inc.

Poxeal Motor Expedites Paper Stock Pumping

Pumping paper stock during flood periods from a low lying stock chest at C. H. Dexter & Sons, Inc., Windsor Locks, Conn., manufacturers of special long fiber papers, has been expedited with the installation of a 5-hp. Poxeal motor.

In the past, the pump motor had to be removed until the flood water had subsided sufficiently to permit the machine to be returned for pumping service.



Now with this Allis-Chalmers epoxy-resin insulated, random-wound, stator-coil motor, pumping continues even during complete submergence of motor and pump. The

motor has operated successfully under 5 to 6 ft. of water for days.

When water recedes from the area, the only maintenance required is to supply new lubricants to the pump and motor.

St. Regis Paper Co.

Appointments

St. Regis Paper Co. announces the appointment of Michael A. Roberts to the new position of manager of Industrial Relations for the northwestern United States. He will represent the company on all industrial relations matters in that area. Beginning Jan. 1, 1960, he will be located at the company's offices at 1019 Pacific Ave., Tacoma, Wash.

Edgar B. Hoppe has been appointed director of European Operations for St. Regis Paper Co. Mr. Hoppe had been European Manager since 1940.

P. H. Glatfelter Co.

Appointments

The P. H. Glatfelter Co. announces three promotions:

Byron C. Miller is promoted to production assistant to the general superintendent.

Paul J. Schmitt was transferred to the pulp division and is assigned the duties of pulp mill superintendent.

Robert D. Owen is appointed engineering assistant to the general superintendent.

Olin Mathieson Chemical Corp.

Special Lightweight Paper

Development of a special lightweight paper for offset duplicating—half the weight of current standard offset duplicating papers—has been announced by George W. McCleary, sales manager for Ecusta Fine Papers, Olin Mathieson Chemical Corp.

Part of the new "Waylite" line, the paper was developed in an accelerated research program to fill a sudden need for lightweight offset duplicating paper created by higher postal rates. It was field tested for 3 months and was introduced nationally on December 1.

In addition to this offset paper, Olin Mathieson recently announced the development of a 17¼-lb. Waylite paper for letterpress printing.

This newest lightweight paper development is part of a research program at Ecusta to produce a line of fine papers having the opacity and printing characteristics of heavier-weight papers now in common use.

Ecusta Paper

Appointments

F. S. Best has been appointed product sales manager for the specialty products department of Ecusta Paper. It was announced by William E. Zimmermann, director of sales and marketing.

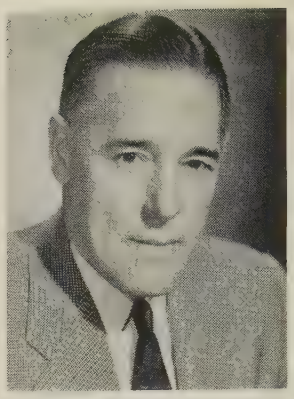
Ecusta Paper, part of the Packaging Division of Olin Mathieson Chemical Corp., is a leading producer of fine lightweight papers, including Bible paper, thin publishing papers, and cigarette paper.

Scott Paper Co.

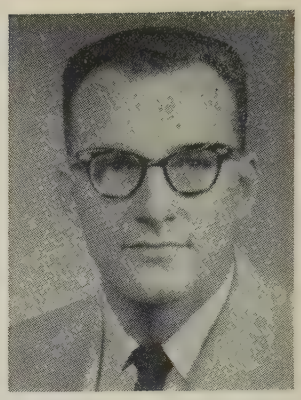
Appointment

Francis W. Plowman, vice-president and director of Scott Paper Co., has been named the company's first director of customer relations.

Mr. Plowman will assist all branches of Scott's sales organization in developing programs and policies best suited to the mutual needs of the company and its customers.



Francis W. Plowman, Scott Paper Co.



Howard S. Rothe, National Gypsum Co.

Rayonier and Hammermill

New Paper Machine

Rayonier and Hammermill will install a second paper machine in the Grays Harbor paper mill at Hoquiam, Wash.

The total capacity of the two paper machines will be in excess of 60,000 tons per year and will provide expanded capacity for a broadened line of fine and printing papers for the growing western market.

It is planned that the project will be completed during the latter part of 1961.

Nekoosa-Edwards Paper Co.

Expansion

Construction is now under way on a pulp storage and shipping building at the Nekoosa mill of Nekoosa-Edwards Paper Co., a leading producer of business and printing papers.

The one-story, 190×120 ft. building will have over 24,00 ft. of storage space and will include two tracks and loading docks to facilitate six cars.

Appleton 5000 f.p.m. Winder

A new 186-in. paper machine winder will be furnished by Appleton Machine Co. for the new no. 2 paper machine at Nekoosa-Edwards' Nekoosa mill.

The winder is designed for 5000 f.p.m. and will be used on shipping and converting rolls of register bond and other writing grades.

Equipped with score cut slitters the unit includes: two 100-hp. drum drive motors driving 21-in. drums, a 100-hp. drag generator for electric tension controlled oscillating unwind, a hydraulic lowering table with weighing scale for wound rolls.

Both the winder and paper machine will be installed and ready for operation by mid-1960:

National Gypsum Co.

Appointment

Howard S. Rothe has been named manager of National Gypsum Co.'s Newburgh, N. Y., paper mill.

At 29, Rothe, who has been acting manager at Newburgh since July 1, becomes National's youngest plant manager.

Marathon, Division of American Can Co.

Water Pollution Control

Marathon, Division of American Can Co., Green Bay, Wis., disclosed that soon no more mill effluents will enter the East River and all used water will be treated to cut discharge of fine solids as well as oxygen demand before being released to the Fox River far downstream.

John E. Goode, plant manager of the firm's Northern mill, announced that construction is now underway of pipelines, pumping stations, and settling basins to cost a minimum of $325,000. The installation, scheduled to operate by July 1, 1960, has been approved by the State Committee on Water Pollution.

International Paper Co.

Appointments

Four promotions within the Southern Kraft Division of International Paper Co., including the appointment of two mill managers, were announced today by C. L. Crain, vice-president and manager of manufacture for the southern Kraft Division.

Eugene E. Ellis, Jr., manager of the company's Natchez, Miss., mill since 1955, was promoted to manager of the Mobile, Ala., mill and James M. Van Hoose, assistant mill manager of the Camden, Ark., mill was promoted to manager of the Natchez mill.

Simultaneously, two production superintendents were promoted to assistant mill managers. They are, for the Mobile mill, Varnado Williams, and for the Natchez mill, Emmett R. Jones.

Mr. Ellis replaces Arthur F. Perkins, Mobile mill manager since January, 1959, whose transfer to the company's division office was announced earlier.

Grays Harbor Paper Co.

Expansion

A new high-speed paper machine and related papermaking facilities, costing in excess of $10 million, will be installed by the Grays Harbor Paper Co. at its paper mill adjacent to the Rayonier pulp mill in Hoquiam, Wash.

The new project, to be completed in late 1961, will increase the plant's capacity from 30,000 to 70,000 tons annually as well as broaden the line of fine and printing papers for the growing western market.

The Grays Harbor Paper Co., formerly known as the Grays Harbor Pulp and Paper Co., is jointly owned by Rayonier Inc. and Hammermill Paper Co. Engineering for the new project will be done on a joint basis with Hammermill supervising paper processing design and equipment specifications and Rayonier supervising plant design and construction.

West Virginia Pulp and Paper Co.

Appointments

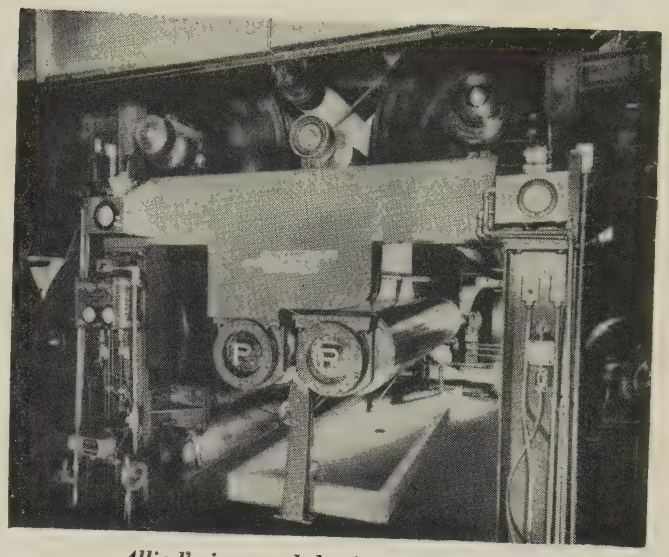
Promotion of Harold A. Sutphen to the position of assistant manager in charge of administration for the Fine Papers Division of West Virginia Pulp and Paper Co. has been announced by John A. Luke, division manager.

Allied Paper Corp.

"Inverted" Horizontal Size Press

Allied Paper Corp., Kalamazoo, Mich., has installed an unusual "inverted" horizontal size press on its no. 10 machine. The installation is considered unusual because: (1) The size press frames are placed upside-down, thus supporting the press in an unconventional "off-the-floor" fashion. (2) The horizontal size press was installed where no press existed before, but even so it only replaces one drier. (3) It is the first known "inverted" horizontal size press installed anywhere.

"This idea was conceived by Ward Harrison, president of Allied Paper Corp. The installation was designed and built by Patton Manufacturing of Springfield, Ohio, who, besides their general line of paper machine equipment, are the recognized specialists in the horizontal size press field. Patton's engineers tackled this project as a challenge in answer to a requirement of removing only one drier, as specified by Ward Harrison.



Allied's inverted, horizontal size press

This "inverted," or as it is more commonly termed by Allied personnel "upside-down" Patton horizontal size press, was installed in Allied's Monarch Mill just prior to the mill's recent reopening. A second "inverted" size press of identical design has just been placed on order with Patton by Allied for its no. 11 machine installation for early 1960. L. B. Owen, vice-president-production, at Allied is satisfied with the first "inverted" installation because, according to Mr. Owen, among other things, this design facilitates simple roll handling and allows for ease of threading and handling of broke. Allied is in an excellent position to evaluate this unusual press, because it has a conventional Patton horizontal size press on its no. 4 machine at the King Division.

Despite the fact that only one drier was removed to make room for the "inverted" press, there is a surprising amount of room around the press and the general access to the unit is completely satisfactory. In making this installation, Patton removed the frame which supported the two driers immediately above the new size press location. The size press frames were then placed upside-down and extended horizontally with two columns coming up to support the inverted framework. This unconventional installation consists of a hydraulically loaded size press unit, so designed with auxiliary rolls that there is a choice of running the web in two different ways after emerging from the press nip. Herbert B. Johnston, vice-president-engineering, of Allied Paper Corp. feels they can obtain a much better draw on their lightweight grades by having short draws, both into and out of the press, as made possible by this installation. Conversely, Allied can run the sheet down under a Teflon covered roll, following which the sheet is guided vertically upward over a Mount Hope roll before making contact with the first top after-drier. When running the short draw, the sheet merely runs horizontally over a Mount Hope roll and into the first bottom after-drier.

According to Wayne T. Crannell, general manager of the Paper Machine Division of Patton Manufacturing, this new installation approach will greatly benefit mills with limited drier capacity, who are being forced by competition to install sizing equipment, but who are unable to either move the after-drier section or to remove three or four driers; either one of which choices having been deemed mandatory.

At the present time, Allied Paper Corp. uses its size presses for conventional starch sizing. The mill hesitates to indicate its future plans on surface sizing.

Another somewhat unusual aspect of this installation is the fact that the sheet leaves the last top drier before the size press and then passes over an air-mounted spring roll before

going directly downward into the nip of the press. There is no doctor on this last top drier, primarily because the pre-drier section rope system guides the tail through the press before making the automatic transfer. The air mounted spring roll consists of a Fawick Corp. air element in which the air pressure may be varied, thus changing the mountings spring rate.

Both of these installations include new size press as well as after-drier machine drives with Falk spiral bevel gear units and Fawick air-operated clutches, driven by taper cone pulleys and nylon belting with roller belt guides.

Appointment

The promotion of Claude Mason to the position of King Mill Conversion supervisor was recently announced by King Mill Manager, A. Mulder.

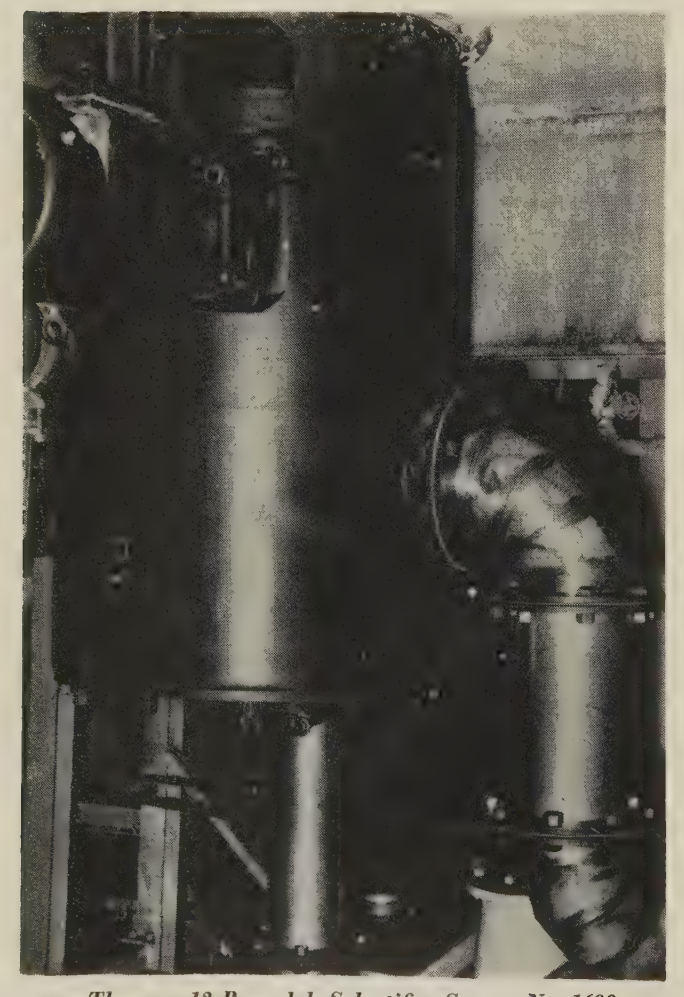
Edward Paulson has been appointed chief maintenance engineer for the Allied Paper Corp.

In making the announcement, H. B. Johnston, vice-president stated that Mr. Paulson will work directly under him and will be responsible for the overall general supervision of the maintenance functions of the King, Monarch and Bryant Mill Divisions.

Black-Clawson Co.

New Selectifier on Sensitized Paper Stock

Selectifier Screen No. 1698, one of the new 12-P models recently designed and built by the Black-Clawson Co.,

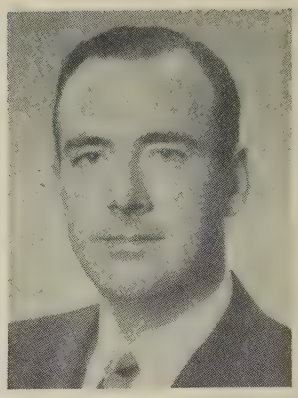


The new 12-P model, Selectifier Screen No. 1698

Shartle Division, has gone into operation at Hartford City Paper Division, 3-M Co., Hartford City, Ind. The 12-P Selectifier screen is a low capacity unit designed to handle a maximum of 1200 g.p.m. at 0.5% consistency. It operates under pressure and completely full of stock at all times.

The new compact, low capacity 12-P model was installed in record time and started up with no difficulties. It is in operation ahead of Hartford City's No. 1 paper machine, a 120 in. fourdrinier producing a high quality, lightweight sulfite grade, used as base stock for the famous "Thermo-Fax" brand copy papers, a 3-M product.

John D. Lynch, Black-Clawson Co.



Robert R. McKellar, Black-Clawson Co.

High Velocity Air Systems Department

The Black-Clawson Co. has established an Air systems Dept. for engineering and marketing its new line of high velocity, air drying equipment. Formation of the specialized unit was announced by Frank Peterson, president of Black-Clawson, manufacturer of an integrated line of pulp and paper mill machinery.

The department is under the joint direction of John D. Lynch and Robert R. McKellar and is headquartered at Foulton, N. Y., in a portion of Black-Clawson's Dilts Division plant.

Crown Zellerbach

Clupak Production Begins

Crown Zellerbach Corp. has begun the first west coast production of Clupak at its Antioch, Calif., mill.

Intended primarily for the multiwall sack and converting grades markets, C.Z.'s. Clupak paper is being run on its no. 1, 260-in. wide machine at Antioch which has an estimated production capacity of approximately 300 tons a day.

Crown Zellerbach is the eighth papermaker to go on line with Clupak paper production since the begining of 1959. International Paper Co., West Virginia Pulp and Paper, Union Bag-Camp Paper Corp., Albemarle Paper Co., Continental Can Co., and St. Regis Paper Co. are already in production in this country and St. Lawrence Corp., Ltd. now produces the paper in Canada.

Appointments

William T. Nye, patent counsel and assistant director of research for the Gaylord Division of Crown Zellerbach, has been promoted to director of patents for C. Z. He will leave the Gaylord research and development offices in St. Louis for C. Z. headquarters in San Francisco in January.

Assistant vice-president F. O. Boylon has been appointed general manager for manufacturing, reporting to E. W. Erickson, vice-president for manufacturing and construction. Mr. Boylon joined Crown Zellerbach in 1948, serving as assistant resident manager of the Ocean Falls, B. C., mill and as resident manager of the Camas, Wash., mill. He is a graduate of the University of Michigan.

R. R. Edwards moves up from product manager for bags and bag paper to assistant vice-president for manufacturing in charge of mill operations in California, Louisiana, Ohio, and New York.

H. H. Wymore, who has been serving as product manager for consumer products, has been appointed assistant vice-president for over-all product management.

Otis D. Hallin becomes vice-president for timber and logging operations. Mr. Hallin succeeds D. S. Denman, senior vice-president for timber, who is retiring December 1 after 33 years with the company.

Mr. Denman, who has been associated with industrial timber operations on the Pacific coast for more than 40 years, continues as a member of the Board of Directors and Finance Committee and will be available to the company as a consultant.

Bowaters Southern Paper Corp.

Appointments

Two officials of Bowaters Southern Paper Corp. have been appointed to new positions.

D. W. Timmis, first vice-president at Calhoun, will become managing director of the Tasman Pulp and Paper Co. Ltd. of New Zealand effective January 1.

V. J. Sutton, vice-president-operations of Bowaters Southern, will become vice-president and general manager of the Tennessee Company. Mr. Sutton will also succeed Mr. Timmis in the latter's post as vice-president of Bowaters Carolina Corp., Catawba, S. C.

The Bowater Paper Corp. Ltd. of London, England, recently acquired a financial interest in the New Zealand company and, in conjunction with Albert E. Reed & Co. Ltd., will be responsible for the management of its operations and will also furnish technical direction for the New Zealand mill. A second paper machine is to be added as part of a new development program. Bowaters Australian sales company has been appointed as the sole selling agents for the products of the Tasman company.

St. Joe Paper Co.

New Semichemical Mill

Plans for constructing a $13,500,000 semichemical, corrugating medium mill in Port St. Joe, were announced by the Florida Gulf Fibre Co., a new company organized to build and operate the new facility.

Roger L. Main, president of St. Joe Paper Co., said that completion is expected by early 1961.

The fully integrated mill will employ approximately 300 persons, and will have a daily capacity of 450 tons of neutral sulfite, semichemical corrugating medium.

Weyerhaeuser Co.

Appointments

Weyerhaeuser Co. announced changes in its officers to be effective Feb. 1, 1960. Norton Clapp of Seattle, well-known Pacific Northwest business leader, has been elected president, succeeding F. K. Weyerhaeuser who will become chairman of the board.

Norton Clapp, chairman of the board since March, 1957, was elected after Weyerhaeuser announced his retirement as president effective February 1.

Mr. Weyerhaeuser has been president of the company since December, 1956, at which time he succeeded his brother, J. P. Weyerhaeuser, Jr., after the latter's death. F. K. Weyerhaeuser served as chairman of the board from March, 1955, until taking over the presidency in 1956.

Southern Land, Timber and Pulp Corp.

$40 Million Pulp Mill

Plans for the construction of a new $40,000,000 sulfate pulp and paper mill in the south were announced by John J. Neely, of Manchester, Ga., president of the Southern Land, Timber and Pulp Corp.

At the same time, Neely announced the appointment of Edward L. Cowan, of Calhoun, Tenn., former vice-president and chief engineer for one of the world's largest paper producing firms, as executive vice-president of Southern Land.

Cowan, whose experience includes 25 years in the engineering and operation of paper producing plants, planned and supervised mill construction involving more than $100,000,000 in capital expenditures in the last four years for the giant Bowaters Corporation at plants in Tennessee, South Carolina, Newfoundland, and Nova Scotia.

The Orr Felt & Blanket Co.

Appointments

Daniel J. Walsh, Jr., has been added to the Orr Felt & Blanket Co.'s felt sales-service staff.

Joe Waters and Dan Walsh will divide the New England territory.

Mr. Walsh spent his life working in the paper field and calling on paper mills.

Great Lakes Paper Co.

Newsprint Production Records

A series of newsprint production records have recently been set in the four-machine paper mill of Great Lakes Paper Co., Fort William, Ont. In one 24-hr. period during the week of November 1 the four Black-Clawson paper machines, running 32-lb. newsprint, established a four-machine, production record of 1193 tons, officials of the company report.

The total includes newsprint from the world's largest paper machine, the 342-in. wide Black-Clawson Hydroflyte fourdrinier which began operation in 1958 and has been successively making and breaking efficiency records in recent months. Operating in a recent 24-hr. period at 1900 f.p.m. and trimming 330 in., the machine produced 383.6 tons.

Even modest increases in speed on the big no. 4 machine and on the other three units will enable the Great Lakes mill to produce more than 1200 tons for 24 hr.

Menasha Wooden Ware Corp.

Expansion

Mowry Smith, president of Menasha Wooden Ware Corp., announced that the firm has finalized its plans to construct a 125 ton neutral sulfite semichemical pulp and paper mill at North Bend, Ore. Construction will commence within a short time and it is anticipated that completion of the facility will be in mid 1961.

Menasha Wooden Ware Corp. has extensive timberland in southwestern Oregon and owns plywood, lumber mill, and wood flour operations at North Bend, Ore., and a corrugated box plant in Anaheim, Calif. The new mill will be a further step in the integration of these West Coast facilities, in that wood residue from the plywood and lumber mills and pulpwood from its and surrounding timber holdings will be used as

E, C. Manders, Menasha Wooden Ware Corp.

raw material and the production of the mill will in part be used at its Anaheim plant. Accordingly, the new North Bend mill will act as an adjunct to the Anaheim box plant just as the company's Otsego, Mich. mill acts to the Menasha, Wis., corrugated box plant.

The corporation has employed E. C. Manders as manager of the new mill. Mr. Manders graduated from the University of British Columbia in 1941 and since that time he has been employed in the pulp and paper industry.

Owens-Illinois

New Box Plant

One of the world's most modern plants for the production of corrugated boxes was officially opened in Chicago by the Owens-Illinois Paper Products Division.

The 224,400-sq. ft. facility, located at 440 East 138th St., near Chicago's southern city limits, replaces a smaller O-I box plant in Chicago's clearing industrial area.

Equipped with a corrugator capable of turning out more than 65 million sq. ft. of corrugated board a month, the plant will produce shipping boxes and other corrugated items covering the entire range of the area's industry from meat packing to heavy machinery.

Six leading executives of the Owens-Illinois Paper Products Division get in the act of the tree planting ceremonies at the companies new Chicago corrugated box plant. From left are Lester R. Edwards, Hugh C. Laughlin, F. W. Wallis, Henry C. Rudy, Edwin D. Dodd, and Leonard T. Moore

Packaging Corporation of America

Appointments

Robert K. Stolz was appointed to the new position of vice-president-forward planning, and John L. Wilson was named vice-president of research and development, of Packaging Corp. of America.

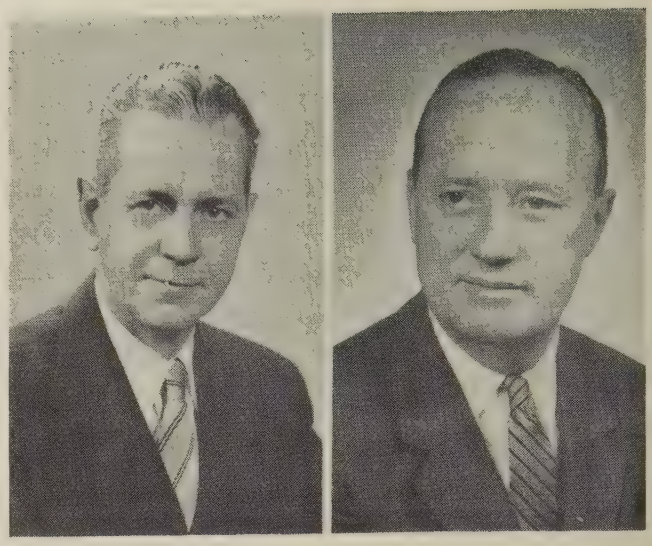
Both men are management veterans of American Box Board Co., of Grand Rapids, Mich., one of the three major divisions of Packaging Corp. The new company was formed recently by the three-way merger of American Box Board, Central Fibre Products Co., of Quincy, Ill., and Ohio Boxboard Co., of Rittman, Ohio.

Nashua Corp.

Appointments

Walter P. McLaughlin has been promoted to sales manager of Nashua Corp.'s Converter Sales Division.

Mr. McLaughlin was formerly manager of sales for the Corrugated Box Industry serving under Carl E. Doane, former sales manager of Converter Sales Division who was recently named to head Nashua's Flexible Packaging Division.



Robert K. Stolz (left) and John L. Wilson, Packaging Corp. of America

Olin Mathieson Chemical Corp.

Dallas Corrugated Container Plant

Start-up of a new corrugated container sheet plant in Dallas was announced by Kent Anderson, product manager, containers, for Olin Mathieson Chemical Corp.'s Packaging Division.

The plant covers approximately 45,000 sq. ft. of floor space and has a production capacity of 5,000,000 sq. ft. of corrugated containers per month. Stewart M. Norris will manage the new Dallas operation.

Among other products of the Packaging Division are kraft paper and board, multiwall shipping sacks, cartons, standard and specialty bags, Olin cellophane, Olin polyethylene, and Ecusta fine papers.

Educational Institutions

Illinois Institute of Technology

The National Science Foundation has awarded grants for basic research to two faculty members at Illinois Institute of Technology. Totaling $8500, the grants went to Robert C. Kintner, acting director of chemical engineering, and Liang N. Tao, associate professor in mechanics.

Dr. Kintner's research is on the velocity of fall of streams and clouds of drops through liquids in finite containers. Its objective is to relate the velocity of fall (or rise) of streams of drops in a liquid field to the performance of a single drop in the same or another fluid field.

A $4400 grant to Dr. Tao is for research on fluid flow between porous rollers. The research deals with experiments using porous rollers for possible use in paper manufacturing.

University of Maine

Pulp and Paper Foundation Awards

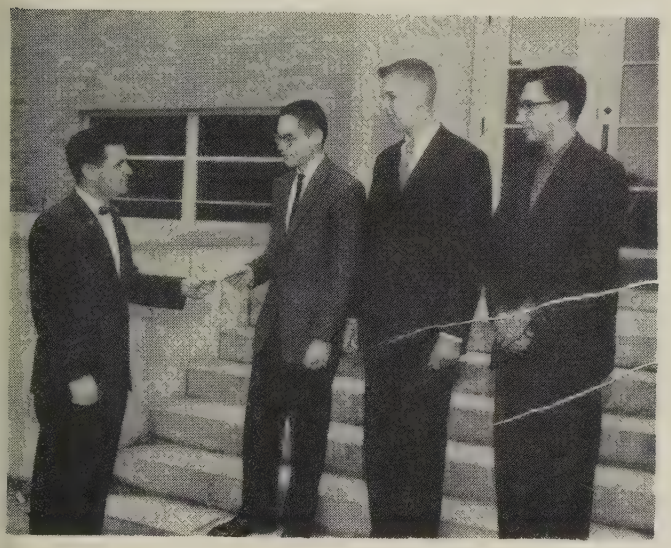
Forty-five, high-ranking students in the pulp and paper technology program were honored at a banquet at the University of Maine, Thursday, November 5. The banquet was sponsored by the University of Maine Pulp and Paper Foundation and honored the students who have received scholarships and grants from the Foundation. Speakers included J. Larcom Ober, of Beverly, Mass., president of the Foundation, and four of the honored students. Mr. Ober is a retired vice-president of the Scott Paper Co. Dean Wes-

ton S. Evans, of the University's College of Technology, presided.

Pulp and paper technology students at the U of M Pulp and Paper Foundation banquet

Lowell Technological Institute

Four Massachusetts students of paper engineering at Lowell Technological Institute, Lowell, Mass., have been awarded $100 scholarships given by 12 paper companies in memory of Geoffrey R. Broughton, former LTI professor.



Left to right are shown Charles J. Higgins, paper engineering instructor, congratulating Ernest A. Tessier of Lowell, '61; John E. Sanderson of Dover, '62; and Frank Zichelle of Fitchburg, '60. The other scholarship recipient, not present for the photograph, is Bernard Coyle of Salem, '61

N. C. State College

PIMA Scholarship Awards

The Southeastern Division of the Paper Industry Management Association awards each year two scholarships amounting to $200 each, these scholarships going to outstanding students in the pulp and paper curriculum at North Carolina State College.

This year's awards, from the William A. Bridges Memorial Scholarship Fund, were made at the annual luncheon of the Pulp and Paper Foundation, held Oct. 23, 1959, by Joseph King, chairman of the Southeastern Division of PIMA to William Harry Peele, Jr., of Plymouth, N. C., a junior student in pulp and paper technology, and to Kindred Pope Magette of Courtland, Va., a senior in the pulp and paper technology curriculum.

The Allied Paper Salesman's Association of New York State's yearly award of $300 to an out-of-state junior student in pulp and paper technology went to Norman Arnold Russell of Raleigh, N. C., a graduate of Needham Broughton High School in this city.

The Institute of Paper Chemistry

Electron Microscopy Lab.

A gift of $30,000 to The Institute of Paper Chemistry from the Louis Calder Foundation for the establishment of a modern electron microscopy and diffraction laboratory has been announced by John G. Strange, president of the Institute.

The Institute's 20-year-old electron microscope was one of the first in the world acquired by an educational and research organization. Through electron microscopy studies valuable contributions have been made to a better understanding of the structure of the papermaking fiber, the modification of that structure, and the use of filler and coating particles. Results of this research in turn would be useful in forest genetics, pulping, coating, and other papermaking operations.

Last year another gift of $50,000 from the Louis Calder Foundation provided a plant biochemistry laboratory at the Institute. This grant has made possible new basic research efforts in fields important to the ultimate technology of the pulp and paper industry.

Legacy of $100,000

A bequest of $100,000 has been received by The Institute of Paper Chemistry from the estate of Marion Bessie Kress for the establishment of the Oscar Kress Foundation to furnish an income for scholarship grants.

Mrs. Kress provided the endowment in memory of her late husband who was the brother of Dr. Otto Kress, Technical Director of the Institute from 1929 until his retirement in 1948.

Dr. Kress, now a resident of Naples, Fla. was the first scientific staff member of the Institute and contributed in numerous ways to its growth as a graduate school and research center.

Industry Suppliers

Electric Steel Foundry Co.

Expansion

Esco now distributes the Hills-McCanna line of diaphragm values in California with stocks located in Los Angeles and the San Francisco bay area. With the addition of California, Esco's distributorship extends to all of the 13 western states.

The distributorship was expanded in 1958 to include Colorado, Arizona, Wyoming and the eastern part of Montana. Esco has been the distributor of this line in Oregon and Washington for the past 4 years.

Hills-McCanna was the first manufacturer of The Saunders Patent Diaphragm valve in 1931. These valves will handle corrosion and abrasion flow problems ranging from air to slurry conveying.

Monsanto Chemical Co.

Expansion

Monsanto Chemical Co.'s Inorganic Chemicals Division has announced the expansion of its line of nonionic surface active agents with the addition of alkyl phenol-based ethylene oxide condensates.

Monsanto already markets alcohol- and mercaptan-based nonionics and for years has been the nation's largest producer of tall oil-ethylene oxide condensates.

Interim production of the dodecyl phenol- and nonyl

phenol-based products is already under way at the company's Kearny, N. J., and Everett, Mass., plants.

Cold Folded Plastic Packages

Nine major carton manufacturing plants have been licensed to produce and supply plastic blanks for the first automatic machine to form sheet plastic into packages without the application of heat or adhesives.

The licensees are:

Robertson Paper Box Co., Montville, Conn.
Schmidt Lithographic Co., San Francisco.
Sample-Durick Co., Chicopee, Mass.
Weyerhaeuser Co., Chicago.

Weyerhaeuser's Boxboard and Folding Carton Division includes these plants:

Ace Carton Co., Chicago, La Puente, Calif., and Middlebury, Ind.
Rochester Folding Box Co., Rochester, N. Y.
Weyerhaeuser Southern, Old Dominion Division, Charlotte, N. C.
Gereke-Allen Carton Co., St. Louis

The new machine development, designated Plasti-Lok, was announced last month by R. A. Jones Co. of Covington, Ky., manufacturer of the machine which cold-folds and locks unscored, die-cut plastic blanks into trays at speeds of 40 to 100 a minute, depending on package size and dimensions.

A second packaging machine based on the cold-folding principle, is the Multi-Fold. It is designed for the setup box plant to form finished containers of various shapes and sizes for such products as wearing apparel and other soft goods, hardware, and toilet goods.

Both machine developments are the result of more than 3 years of cooperative effort by Monsanto Chemical Co., Charles E. Palmer & Associates and R. A. Jones Co., using Polyflex oriented styrene sheeting manufactured by Plax Corp. of Hartford, Conn. The continuing integrated program by a resin producer, a machine developer, machine manufacturer, and a material supplier is believed to be the first of its kind in the packaging industry.

Air Reduction Sales Co.

New Plant at Baton Rouge

Air Reduction Sales Co. is constructing a new air separation plant at Baton Rouge to supply high purity liquefied industrial gases to the Gulf State area.

The new $2,000,000 plant will have a production capacity of 30 tons of liquid oxygen, nitrogen, and argon per day. The installation, the newest in Airco's growing network of plants, will serve all of the industries in the Gulf State area, particularly the expanding petroleum and petrochemical producers. Liquid oxygen produced at the plant will also be made available to the area's hospitals through the Ohio Chemical & Surgical Equipment Co., a division of Air Reduction Co., Inc.

U.S. Testing Co., Inc.

Russell H. Armitage, manager of the Paper Technology Division of the United States Testing Co., announced the publication of a new price list covering the Testing Co.'s services in the paper field.

In addition to the standard test facilities, the Paper Laboratory also has available to Gelboflex tester, Martin Sweets' Neenah expansimeter, Sentinel heat seal tester, Gurley-Hill or Bekk Smoothness testers and the Thwing-Albert Handle-O-Meter.

Copies of the new price list may be obtained by writing to the Paper Technology Division, United States Testing Co., Inc., 1415 Park Ave., Hoboken, N. J.

Chain Belt Co.

Merger with Carrier Conveyor

Merger of the Carrier Conveyor Corp., the country's leading manufacturer of vibrating conveyors, and its subsidiary, General Industries, both located at 211 N. Jackson St., Louisville, Ky., into Chain Belt Co., Milwaukee, Wis., was announced by E. M. Rhodes, vice-president of Chain Belt's industrial section.

Carrier Conveyor Corp. designs and manufactures a unique line of natural frequency vibrating conveyors, feeders, and related equipment for the handling and processing of bulk materials such as ore, cement, chemicals, ashes, coal, sand and gravel, food, and pharmaceuticals.

Hercules Powder Co.

Expansion

Hercules Powder Co. announced plans for a multimillion dollar expansion of its West Coast facilities at Hercules, Calif. The expansion includes the construction of new manufacturing facilities for the production of methanol, formaldehyde, urea-formaldehyde concentrates, and slow nitrogen release urea-form for fertilizer applications.

John M. Martin, general manager of Hercules Explosives Department, said that construction of the new facilities for producing the urea-formaldehyde composition would be completed by mid-1960, with methanol-formaldehyde facilities completed in early 1961.

The new construction will provide facilities for the production of 8,000,000 gal. of methanol per year, 50 million pounds of formaldehyde, and 11,000 tons of urea-formaldehyde compositions.

Currently there is no methanol production in the Western States. Methanol is used principally in the manufacture of formaldehyde. It is also used in the manufacture of other chemicals, plastics, coatings, drugs, solvents, and antifreeze.

International Department

The creation of a new operating department for Hercules Powder Co., to be known as the International Department, was announced by Albert E. Forster, Hercules president.

The newly created department will include the work of the present Export Department, and will have equal status with the company's six industrial departments—Cellulose Products, Explosives, Naval Stores, Paper Makers Chemical, Synthetics, and Virginia Cellulose.

Henry A. Thouron has been named general manager of the International Department. Mr. Thouron, a member of the company's Board of Directors, has been general manager of Hercules Synthetics Department since 1955.

Appointments

Donald H. Sheffield, of West Chester, Pa., has been appointed general manager of Hercules Powder Co.'s Synthetics Department.

Mr. Sheffield, who has been assistant general manager of Synthetics since December, 1955, will succeed Henry A. Thouron.

E. D. Jones Corp.

Sveen-Pedersen Acquisition

The E. D. Jones Corp., Pittsfield, Mass., a member of the Beloit group, announced the acquisition of the complete North American Assets of the Sveen-Pedersen Sales Corp. of Long Island City, N. Y.

Sveen-Pedersen Corp., founded in 1951, has directed the sales, engineering, and manufacturing of Sveen-Pedersen Flotation Savealls in the United States. Since its introduction to the U. S. paper mills in 1938 the Sveen-Pedersen

H. E. Gorton, P. B. Hamilton, and H. C. Weeks (from l. to r.), Dennison Manufacturing Co.

Saveall has become universally accepted in the paper industry as a leader in white water clarification and stock reclamation.

Dennison Manufacturing Co.

Appointments

To complete the reorganization of the company to a divisional setup, Dana C. Huntington, president of Dennison Manufacturing Co., Framingham, Mass., announced recently that Howard E. Gorton, Philip B. Hamilton, and Howard C. Weeks have been elected to the newly created posts of divisional vice-presidents. Mr. Gorton becomes vice-president in charge of the Machine Systems Division and the Industrial Products Division; Mr. Hamilton, vice-president in charge of the Resale Products Division and the Holiday Division; Mr. Weeks, vice-president in charge of the Gummed Paper Division, the Box Division, and the Therimage Division.

Bernhardt L. Sauter has been appointed Industrial Products national sales manager and Frank T. Gerould has been appointed Machine Systems national sales manager. Their headquarters will be at Framingham.

It was also announced that Matthew P. Clarke has been appointed merchandise manager of Machine Systems.

Dorr-Oliver, Inc.

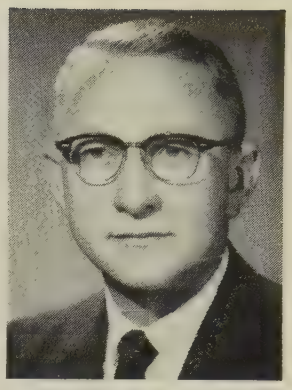
Appointments

The Board of Directors of Dorr-Oliver Inc., appointed J. D. Hitch, Jr., president, to fill its vacant board chairmanship and elected L. R. Boling, currently executive vice-president, to succeed Mr. Hitch as president. Both appointments effective on Dec. 1, 1959, with Mr. Boling continuing as chief executive officer of the internationally known engineering concern. John V. N. Door and Wm. L. Oliver will continue as honorary chairman and vice-chairman of the board respectively.

New Jersey Zinc Co.

Appointments

Robert W. Munson has been appointed Eastern District assistant sales manager by The New Jersey Zinc Co. His principal assigment will be the supervision of the company's pigment sales activities in the southern area of the Eastern District.



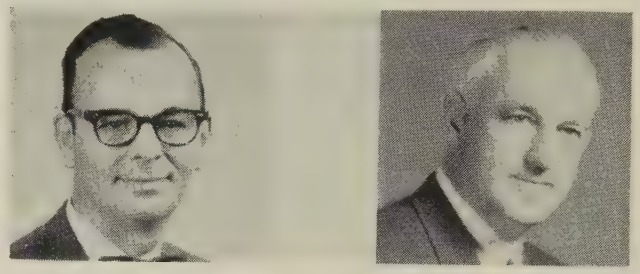
Robert W. Munson, The New Jersey Zinc Co.

Link-Belt Co.

Appointments

Wayne J. Mitchell has succeeded Lester T. Graham as Link-Belt Co.'s district manager at Portland, Ore. Mr. Graham is retiring after 42 years with Link-Belt Co. and its Pacific Coast predecessor organization.

Mr. Mitchell joined Link-Belt Co. at its Minneapolis plant in 1948, as a designer and later as assistant to the chief engineer. He was transferred to the Seattle plant in 1951 as assistant chief engineer, and he was appointed sales engineer at the Portland district sales office in 1956. He is a civil engineering graduate of Iowa State College.



Wayne J. Mitchell (left), and Lester T. Graham, Link-Belt Co.

Pennsalt Chemicals Corp.

Appointments

In a move to place more emphasis on its marketing activities, Pennsalt Chemicals Corp. has created the new office of vice-president of marketing. Albert H. Clem, experienced executive in Pennsalt sales and service, has been named to the post.

Replacing Mr. Clem as general manager of the Chemical Specialties Division is George R. Lawson, formerly director of marketing for Pennsalt's Industrial Chemicals Division.

Dow Chemical Co.

Plant on West Coast

The Dow Chemical Co. announces it will construct a phenol plant with a capacity of 36 million pounds per year in the Pacific Northwest.

The plant will be built at a location still to be selected on deep water at Puget Sound, or in the Longview, Wash., or the Portland, Ore., area. Options on some sites have been secured and others still are under study.

Dow Chemical Co. (U. K.)

The British subsidiary of The Dow Chemical Co.—Dobeckmum Britain Ltd.—has changed its name to Dow Chemical Co. (U. K.) Ltd. and broadened the scope of its activities.

The company will continue to handle the manufacture and sale of Lurex, a nontarnishing metallic yarn, produced in Windsor, Berkshire. In addition, it will take over direct sale of Dow-produced packaging films such as Trycite (polystyrene film), Saran Wrap (vinyl-vinylidene chloride copolymer film), and polyethylene film. It will also supervise the

J. D. Hitch, Jr., Dorr-Oliver Inc.

Lloyd R. Boling, Dorr-Oliver Inc.

sale of Dow U. S. and Canadian-produced industrial chemicals, plastics, and magnesium in the United Kingdom.

In the latter line of products, the company will continue to use the services of appointed distributors. R. W. Greeff & Co. Ltd. will continue as principal distributor handling chemicals, coatings, raw materials, and magnesium. British Resin Products Ltd. is the distributor for certain plastics molding materials; W. J. Fraser & Co. Ltd. for Dowtherm, a heat exchange system; and Union Glue & Gelatin Co. for Dowicide, industrial preservatives.

Appointments

Macauley Whiting has been appointed general manager of Dow's Midland Division, it was announced by Leland I. Doan, president of the Dow Chemical Co.

He succeeds the late William H. Schuette, vice-president of Dow and general manager of its Midland Division, who died unexpectedly Sunday, November 8.

Robert C. Witt has been named product manager for the sale of latexes to the paint industry and of coatings materials for building products, the Dow Chemical Co. announces.

Witt, formerly a coatings salesman with the Dow Chicago office, will take up his new duties in Midland, Mich., Dow headquarters. He succeeds H. Winston Haskell who has been transferred to Dow Chemical International Ltd. S. A., as project sales manager in the Mediterranean area, with headquarters in Zurich, Switzerland.

Stauffer Chemical Co.

Expansion

Stauffer Chemical Co. has completed plans to build a major research center at Richmond, Calif. It will be located on a 10-acre tract recently acquired by the company adjacent to the present Richmond plant and research laboratories.

Construction of the first unit of the new research center will begin in January. Completion of this initial laboratory, which will cost about $1.6 million, is scheduled for early 1961.

The existing research facilities at Richmond will be used to expand process development and pilot plant activities.

Nalco Chemical Co.

Appointments

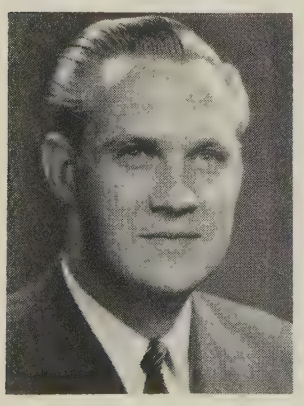
Herbert S. Johnson, Jr., has been made vice-president in charge of marketing for Nalco Chemical Co.

Leonard R. Robinson, Jr., was named to replace Johnson as manager of the industrial division.

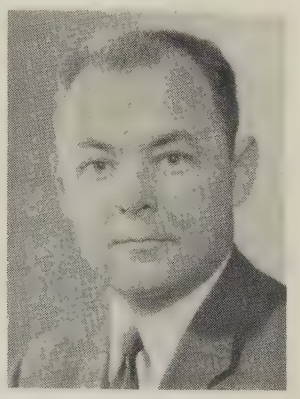
Beckman Instruments, Inc.

Appointment

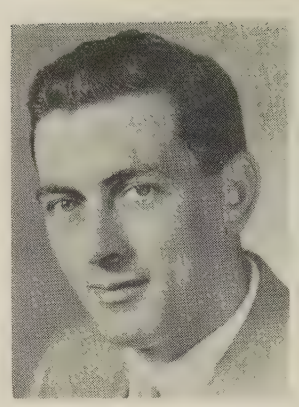
Stuart N. Davidson has been appointed as corporate contracts administrator for Beckman Instruments, Inc., Fullerton, Calif., is announced by William W. Wright, financial vice-president.



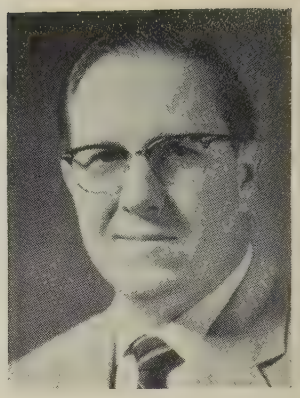
Herbert S. Johnson, Jr., Industrial Division, Nalco Chemical Co.



Leonard R. Robinson, Jr., Industrial Division, Nalco Chemical Co.



Kenneth E. Cosslet, U. S. Industrial Chemicals Co.-International



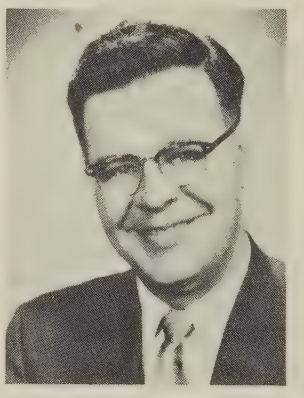
Howard W. Woodham, U.S. Industrial Chemicals Co.-International

U. S. Industrial Chemicals Co.

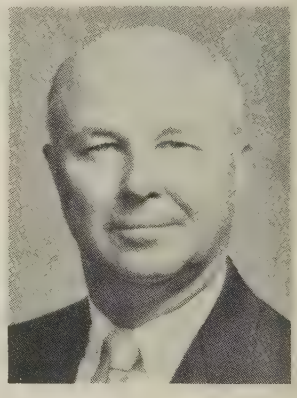
U.S.I.-International

A new company known as U. S. Industrial Chemical Co.-International has been formed. The company will provide sales assistance to European representatives handling U.S.I.'s Petrothene* polyethylene, as well as intensified technical service to European polyethylene processors.

The company, officially incorporated as Sales and Development Co. of National Distillers and Chemical Corp. (International) S.A., will be known to the trade as U.S.I. International. Temporary headquarters were opened in September, 1959, at Kirchenstrasse 13, Zug 1, Switzerland. Permanent headquarters and a technical service laboratory will be constructed on land just purchased in Baar, Canton of Zug.



J. A. Putnam, U.S. Industrial Chemicals Co.

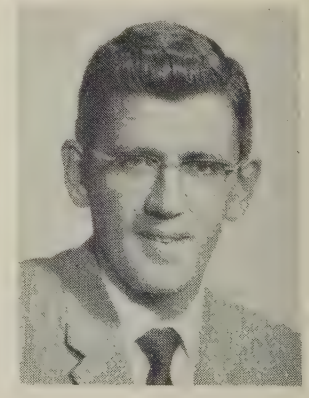


Roy A. Parker, U.S. Industrial Chemicals Co.

Kenneth E. Cosslett, U.S.I.'s assistant export manager, has been named sales manager of "U.S.I.-International" and Howard W. Woodham will be manager of the technical service laboratory. Approximately 12 people will be employed by the new company by early 1960.

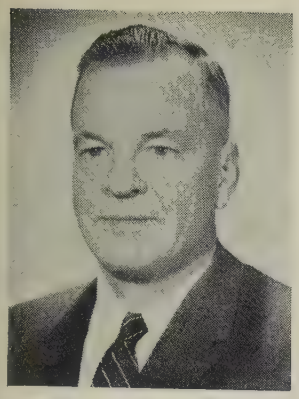
Appointments

John A. Putnam has been appointed manager of the Atlanta office of U. S. Industrial Chemicals Co., Division of National Distillers and Chemical Corp.



Joseph G. Longstreth, U.S. Industrial Chemicals Co.

* Registered trademark.



Albert C. Brooks, Baltimore Sales Div., U. S. Industrial Chemicals Co.

James F. Whitescarver, Baltimore Sales Div., U. S. Industrial Chemicals Co.

Roy A. Parker has been named Kansas City Sales Division manager for U. S. Industrial Chemicals Co., Division of National Distillers and Chemical Corp.

Mr. Parker has been associated with U.S.I. for the past 33 years and, prior to his new assignment, was manager of the company's Kansas City, Missouri Sales Office. He is a past president and member of the Paint, Varnish and Lacquer Association, a member of the Oil Men's Club, and a member of the Midwest Chemical Sales Club, all of Kansas City.

Joseph G. Longstreth has been named manager of the St. Louis, Missouri Sales Office of U. S. Industrial Chemicals Co., Division of National Distillers and Chemical Corp.

Mr. Longstreth has been a sales representative for the company in the St. Louis area since he joined U.S.I. in 1955. He was born in St. Louis and is a graduate of Washington University where he obtained an A.B. degree in Chemistry and Zoology. He is a member of the Society of Plastics Engineers and the Associated Drug & Chemical Industries of Missouri.

Albert C. Brooks has been named manager of the Baltimore Sales Division for U. S. Industrial Chemicals Co., Division of National Distillers and Chemical Corp. Alden R. Ludlow, Jr., vice-president in charge of sales, announced the appointment. Mr. Brooks succeeds James F. Whitescarver who has retired.

Wyandotte Chemicals Corp.

Appointment

Lewis M. Ludlow has been named manager of the Pacific District for Wyandotte Chemicals Corp., Michigan Alkali Division, replacing J. M. Norcott who has resigned.

Sulphur Export Corp.

Appointments

Election of Peter A. Dimitri and Ernest A. Graupner as vice-presidents of the Sulphur Export Corp. has been announced by B. C. Hughes, chairman. Sulphur Export Corp. (Sulexco) handles all marketing outside of North America and Cuba on behalf of all domestic producers of Frasch sulfur. It was established in May, 1958, under the Webb-Pomerence Act which is designed to encourage the expansion of American exports.

Scott Testers, Inc.

Appointments

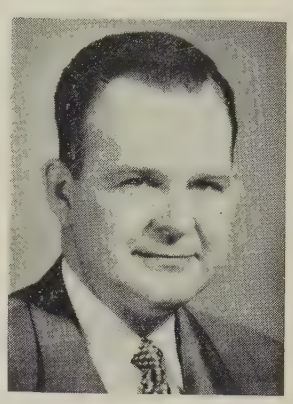
Walter E. Anderson has been appointed as sales engineer of Scott Testers, Inc. The firm manufactures physical testing equipment for textiles, paper, plastics, wire, leather, and other basic industries. Mr. Anderson will be responsible for sales of Scott testers throughout the continental United States.

A. E. Staley Manufacturing Co.

Appointment

Natt K. Hammer has been named Industrial Products Sales Manager of the A. E. Staley Manufacturing Co., Decatur, Ill., corn and soybean processors.

Walter E. Anderson, Scott Testers, Inc.



N. K. Hammer, A. E. Staley Mfg. Co.

Graver Water Conditioning Co.

Appointments

Marvin Lane, technical director of Graver Water Conditioning Co., N. Y., a division of Union Tank Car Co., has been named general manager of that division.

He succeeds H. T. Sulcer, who recently resigned.

Graver Water is actively engaged in the design and development of new products and systems for industrial and municipal water treatment, industrial waste treatment, boiler feedwater treatment, and chemical processing.

Nuodex Products Co.

Appointment

Joseph P. Palumbo has been appointed manager of the plastics industry division of Nuodex Products Co., Division of Heyden Newport Chemical Corp., Elizabeth, N. J.

The division was created because of the growth of the company's line of vinyl stabilizers, organic peroxide catalysts, accelerators, and fungicides.

Diamond Alkali Co.

Appointment

Martin F. Wilkerson, branch manager of Diamond Alkali Co.'s Southwest District Sales Office, has been promoted to sales manager, Chlorinated Products Division, Cleveland, Ohio.

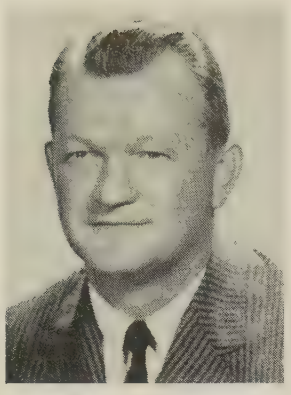
At the same time, Robert L. Walker, salesman for the Southwest District Sales Office was promoted to the branch manager's position, replacing Wilkerson.

Realignment of responsibilities involving three key personnel in Diamond Alkali Co.'s Electro Chemicals Division was announced by A. B. Tillman, operations manager of the division.

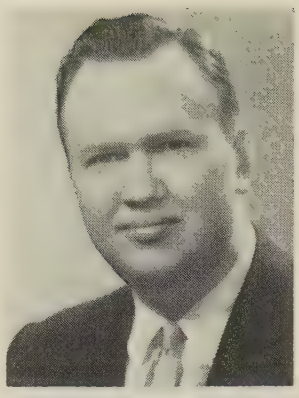
John H. Fonner, Jr., transfers from Diamond's Muscle Shoals, Ala., plant to the Edgewood, Md., plant as assistant plant manager.

Erich G. Schlaile moves from the Edgewood plant to Diamond's Deer Park, Tex., plant as a process engineer.

Robert S. Stark also transfers from the Edgewood plant to the research and development department at Painesville, Ohio.



J. R. Perry, Fuller Co.



Gene White, John W. Bolton & Sons, Inc.

Fuller Co.

Expansion

As part of a general revision and enlargement of its representation in the south and southwest, Fuller Co. has opened a district office in Houston, Tex.

Designed to serve the increasing demands of customers in the area, the new office will be managed by Joseph R. Perry.

General Chemical Division, Allied Chemicals

Appointments

Albert B. Connelly has been appointed manager of the Birmingham, Ala., sales office of Allied Chemical's General Chemical Division. He succeeds John T. Wiegand, who has been named manager of the Los Angeles, Calif., office.

John W. Bolton & Sons, Inc.

Appointment

John W. Bolton & Sons, Inc., Lawrence, Mass., announces that Eugene J. White of Conshohocken, Pa., now represents the Bolton Co., selling their full line of machine knives throughout southern New Jersey, Pennsylvania, Maryland, Delaware, and Washington, D. C.

Bolton machine knives find their major uses in the pulp and paper, graphic arts, plastics, woodworking, and metalworking industries.

Babcock & Wilcox Co.

Appointment

Robert A. Barr has been elected a vice-president of The Babcock & Wilcox Co. by the board of directors, and will assume charge of the Refractories Division.

Mr. Barr will replace James E. Brinckerhoff, vice-president in charge of the Refractories Division, who is retiring December 31 after 40 years of service.

Robert A. Barr, Refractories Div., The Babcock & Wilcox Co.

M. M. Young, Dietz Machine Works

Dietz Machine Works

New Management

A group headed by Michael M. Young has purchased the Dietz Machine Works, Inc. of Philadelphia. Dietz has been an important supplier of machinery for the paper converting industry for 60 years.

Mr. Young, new president of Dietz Machine Works, Inc., was one of the founders and a vice-president of Wabash Manufacturing Co., Baltimore, for the last 12 years. Prior to that time he served as chief production engineer for Bendix Aviation Corp.

Reginald Couzens, who has been with Dietz for a number of years, has been appointed vice-president in charge of Engineering. Mr. Couzens had previously been associated with Scott Paper Co., Chester, Pa. as a machine design engineer.

National Starch and Chemical Corp.

New Resins

A new vinyl acrylic copolymer solution having exceptional utility for laminating or formulation of pressure-sensitive adhesives is now being offered by the Resin Division of National Starch and Chemical Corp.

The Resyn, designated 26–2404, is tacky and is claimed to show specific adhesion to most cellulosic and synthetic films such as cellulose acetate, "Mylar" polyester film,* vinyl, and foil. Resyn® 26–2404 is one of several new vinyl acrylic copolymer emulsions which National has developed within the past year.

A particular advantage of Resyn® 26–2404 is said to be its applicability in the formulation of packaging, pressure-sensitive, and specialty adhesives, as well as a printing ink vehicle and in the laminating of Mylar, cellophane, nylon, polyethylene, saran, paper, and foil. In addition to providing good adhesion to all these type surfaces, the new resin can be thinned easily with inexpensive solvents, provides excellent heat and light stability, and is compatible with a variety of modifying resins.

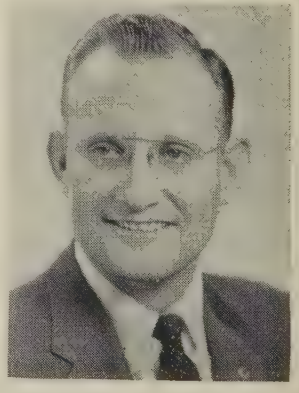
Other resins which have been developed recently as a result of the company's program of basic research in vinyl acetate polymerization include a vinyl acrylic resin for latex paints which is compatible with zinc pigments, Resyn® 2243; a resin which offers unique properties for formulation of self-polishing and rebuffable floor waxes, "Glitter" 2700; and a resin which is now being tested by several of the country's largest packers of aerosol hair sprays, Resyn® 28–1310.

Data on all of the above may be obtained by writing to the Resin Division, National Starch and Chemical Corp., 750 Third Ave., New York 17, N. Y.

Appointment

Leonard J. Wood has been appointed product manager of the Paper Division at National Starch and Chemical Corp.

Mr. Wood has been with National since 1946, starting as a chemist in the paper service and development dept. In 1953 he was transferred to the New England sales office, in charge of adhesive and starch technical sales. He was made supervisor of paper technical service in 1955 and in 1957, paper development and service manager.



L. T. Wood, National Starch and Chemical Corp.

*DuPont's registered trademark.

E. Leitz, Inc.

New Micro Laboratory

A new microscope laboratory designed to provide unique services to science and technology on current problems in optical techniques has been established at E. Leitz, Inc., New York, N. Y., it has been announced by Emil G. Keller, vice-president.

The American firm distributes miscroscopes and many other scientific instruments made by Ernst Leitz, GmbH., Wetzlar, Germany, century-old leader in the field.

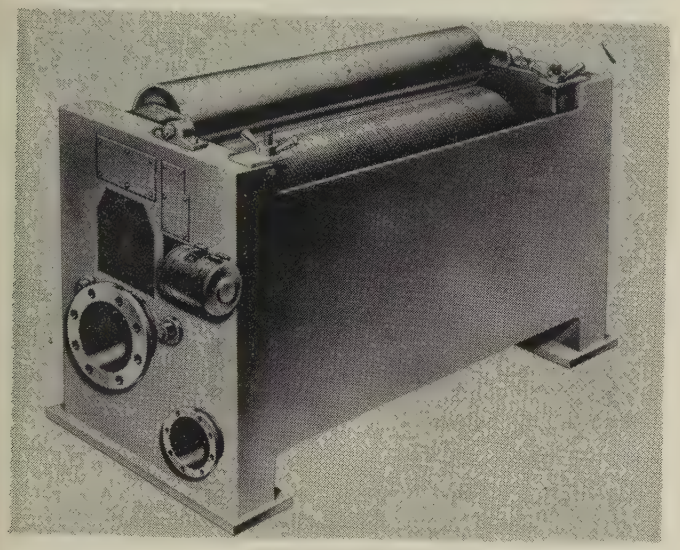
Peter H. Bartels, specialist in applied optics, has come to the United States to join the firm and to direct the laboratory.

The laboratory is located at 468 Park Ave. South and includes a comprehensive collection of scientific instruments. Considerable information as well as the latest optical data available from sources here and abroad can be obtained.

Koehring-Waterous Ltd.

The Gravi-Decker, a new pulp decker with a unique free-flow cylinder mold, is being manufactured by Koehring-Waterous Ltd., Brantford, Ont., a subsidiary of Koehring Co.

The Gravi-Decker's cylinder mold stays clean longer because it has no central shaft, spiders, or winding wires to cause a pile-up of loadings; cleaning can be accomplished without removing the mold from the vat.



The Gravi-Decker

The facing wire in the new decker is mounted on a corrugated stainless steel drum, eliminating the need for a backing wire. Drainage holes in the flanks of the corrugations provide a free area greater than that of the facing wire, so that the drainage rate of the mold is limited only by that of the facing wire.

The built-in master panel—which controls drive, pickup roll lifting and lowering, doctor pressure, drive gear shower, and end seal water feeds—can be removed and mounted separately if mill arrangement requires it.

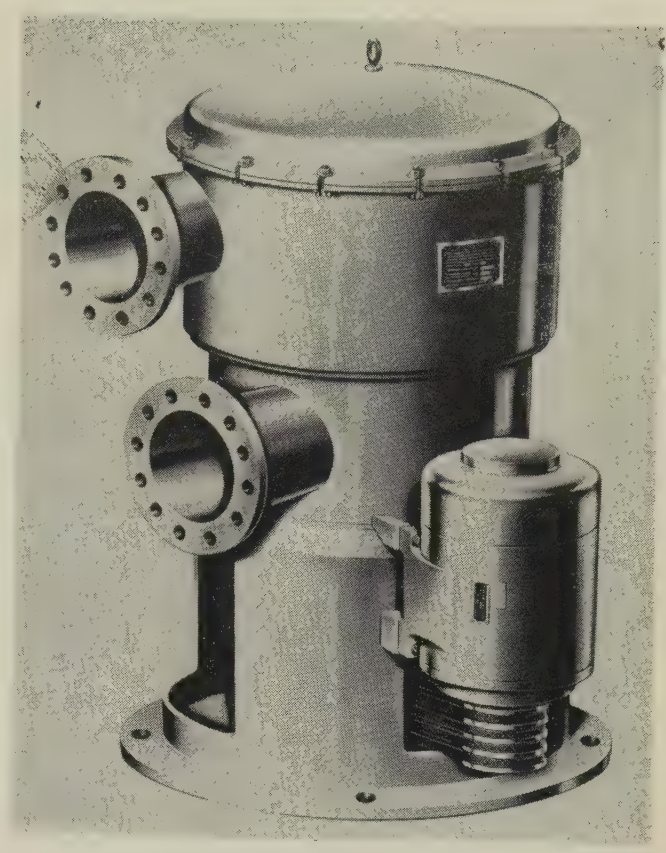
All parts of the decker in contact with the stock not made of stainless steel, bronze, or plastic are rubber-covered.

Further information available from Koehring-Waterous Ltd., P. O. Box 607, Brantford, Ont., Canada.

Vertifoil Pulp Screen

The Vertifoil, a new pulp screen that adjusts to any pulp while running, has been announced by Koehring-Waterous Ltd., the originator of foil-type screens.

A single, variable control sets the screen's special foils to handle the requirements of all pulps—groundwood, sulfite, krafts, etc. Screen changing is unnecessary.



Vertifoil pulp screen

These special foils combine the best characteristics of both aerofoil and hydrofoil sections. They clear screen plates efficiently with minimum power, and without forcing dirt particles or shive through screen perforations with accepted stock.

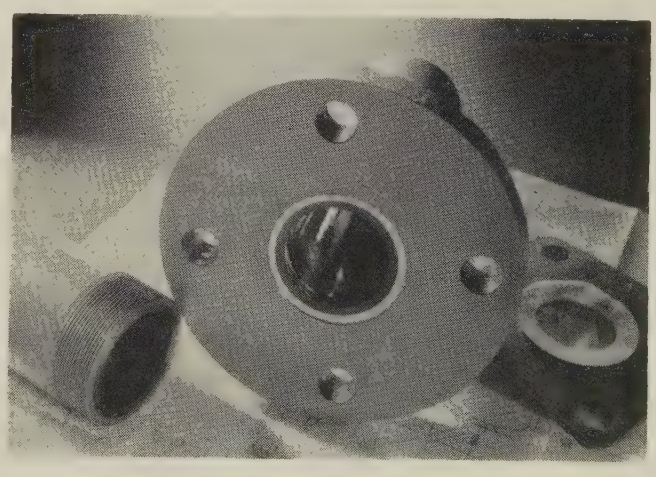
All parts of Vertifoil in contact with stock are built of stainless steel. Drive shaft is isolated from stock by labyrinths and clear water seals. Trash collecting chamber protects screen form damage by debris entering with stock.

Further information available from Koehring-Waterous Ltd., P. O. Box 607, Brantford, Ont., a subsidiary of Koehring Co.

The Pfaudler Co.

Glasteel Pipe

A new Pfaudler Glasteel pipe (glass inside, steel outside) can be field-cut to required lengths, and has more than twice the glass thickness of previous glass-lined pipes.

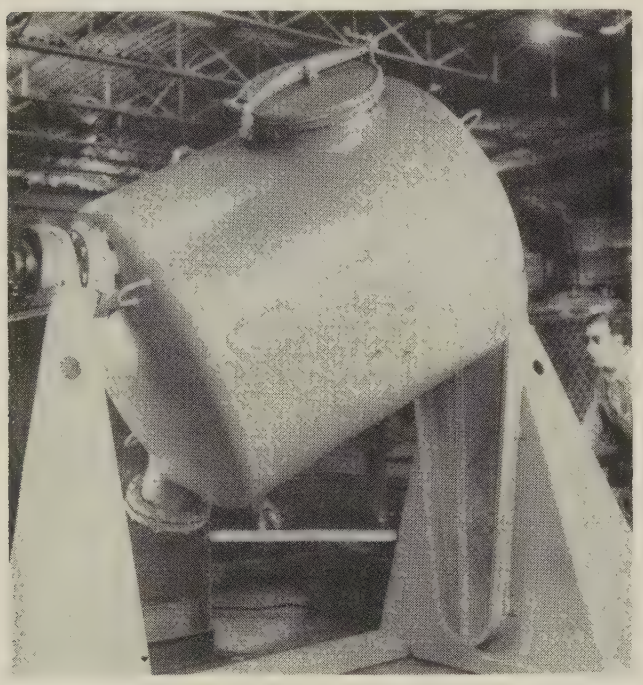


Pfaudler's Glasteel pipe

Rated 150 p.s.i. and available in $1^1/_2$, 2, and 3 in. diameters, Pfaudler F-C Glasteel Pipe* (F-C for "Field Cut") offers wide application in highly corrosive services (all acids, except HF, to 350°F. and even to 450°F. depending on concentration, and mild alkalies at moderate temperatures). It is also ideal for those applications where freedom from product contamination or adhesion is required. According to Pfaudler, corrosion resistance and abrasion resistance, which are the primary determinant of service life, are approximately doubled because of the substantially thicker ($^1/_8$ in.) boro-silicate glass lining.

Glasteel Chemo-Blender

The Chemo-Blender is the first unjacketed, glassed-interior, dry product blender ever produced and is expected to find particularly wide acceptance in the pharmaceutical and fine chemical industry where the characteristics of Glasteel (noncontaminating, high adhesion and corrosion resistance,



Dry product, Glasteel blender by Pfaudler

easy cleaning) are particularly desirable. All interior surfaces have an extremely smooth, fired, glass finish. Products will neither adhere nor build up, and at the conclusion of a cycle all surfaces can be cleaned by simply flushing with water.

Further information may be obtained from the Pfaudler Division of Pfaudler Permutit Inc., 1098 West Ave., Rochester 11, N. Y.

Kimble Glass Co.

Square Volumetric Flask

A square-shaped, borosilicate, volumetric flask is now being introduced by Kimble Glass Co., subsidiary of Owens-Illinois.

These new space-saving flasks are especially designed to pack more closely on shelves, in refrigerators and in cabinets. They come in five sizes: 100, 250, 500, 1000, and 2000 ml. The 1000-ml. size covers 38% less shelf area than a conventional flask of equal capacity.

Kimble's entire line of volumetric flasks is now available in hard glass.

Appointments

Carl J. Dunivin, of Los Angeles, vice-president of National

*Patent Applied for.

Container Corp. of California, has been appointed as both general manager of the company's Los Angeles plant and assistant to the president in charge of Southern California operations was announced by George J. Schneider, president and general manager of the Owens-Illinois subsidiary.

William J. Prudler, of Oakland, Calif., vice-president of National Container of California as well as general manager of its Oakland plant, also has been given the additional title of assistant to the president in charge of Northern California operations.

Cochrane Corp.

Water Demineralizer

A packaged demineralizer designed for quick installation has been developed by Cochrane Corp. On-site erection consists of setting the unit in place and connecting to air and raw water supply, treated water outlet, and open drain. Only electrical connection required is for a conductivity controller, which uses 110-v., 60-cycle current from a standard lighting outlet.

Designated as the Uni-Pac "M" series, models of the new demineralizer are available in capacities from 2100 to 13,200 gal. per hr. using raw water with 10 grains per gallon total exchangeable anions.

A brochure describing the new packaged demineralizer is available from the Cochrane Corp., 17th St. below Allegheny Ave., Philadelphia 32, Pa.

Water Softening Manual

A recently published booklet by Cochrane Corp. describes sodium zeolite process of water softening and gives information on selecting and sizing of equipment. Automatic versus manual controls and calculations of softening capacity are discussed. The more frequently used zeolite materials, both siliceous and nonsiliceous, are described. Illustrations and cutaway drawings show typical treatment systems.

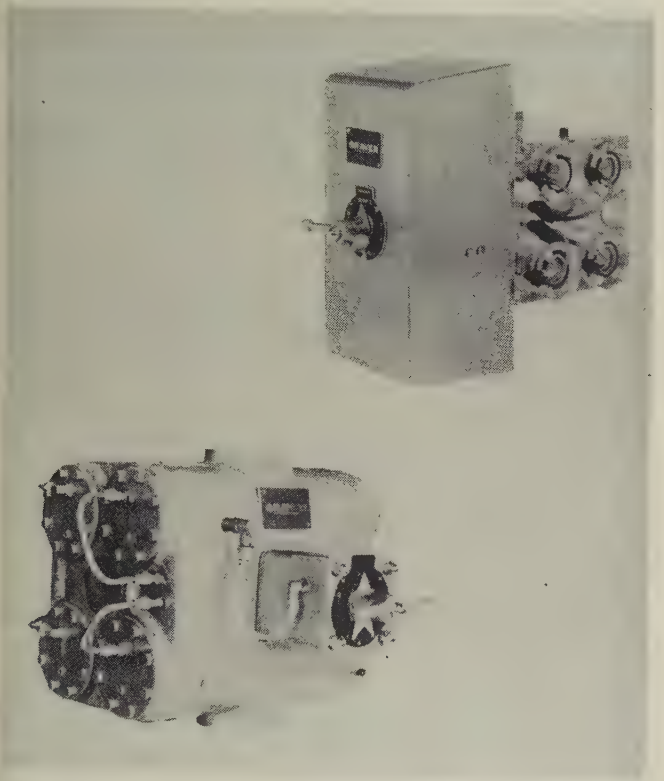
Copies of the new publication "Sodium Zeolite Softners"

(#4520) may be obtained without charge from Cochrane Corp., 17th St. below Allegheny Ave., Phila. 32, Pa.

Graver Water Conditioning Co.

Single Control Valve

A single control valve for directing the flow of liquids in zeolite softeners, ion exchange units and filters has been introduced by Graver Water Conditioning Co., New York. Called the Monotrol®, the valve is designed to perform the functions of a nest of individual valves while providing greater ease in operation and maintenance. It is available in cast iron or in acrylic plastic for corrosive service, and can be operated manually or automatically.



Monotrol valves: top, plastic unit with automatic pilot; bottom, cast iron unit with manual pilot

Features of the valve include pilot control, positive hydraulic pressure actuation, and easy maintenance. All internal moving parts can be readily removed for inspection and service without dismantling the connecting piping or removing the valve from the unit on which it is being used. In addition, the design provides for low pressure loss during operation and no contamination of effluent with influent.

For further information, contact: R. S. Lewis, Manager, Sales Promotion, Graver Water Conditioning Co., 216 West 14th St., New York 11, N. Y.

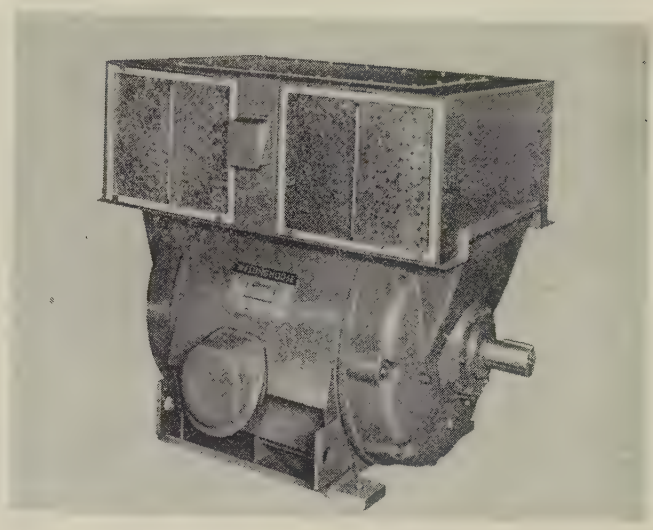
Westinghouse Electric Corp.

New Press-Drive Motors

New press-drive motors (type WPD) from 1 to 300 hp., designed for metal cutting and forming machines, are available from the Westinghouse Electric Corp. These motors are all high slip, high-starting torque, and low-starting current, and are recommended wherever extremely dirty atmosphere or high concentration of oil moisture or chemicals are present.

In frames 182 to 8120, these motors are constructed in four general types—drip proof, totally enclosed fan-cooled, explosionproof, and forced-ventilated motors.

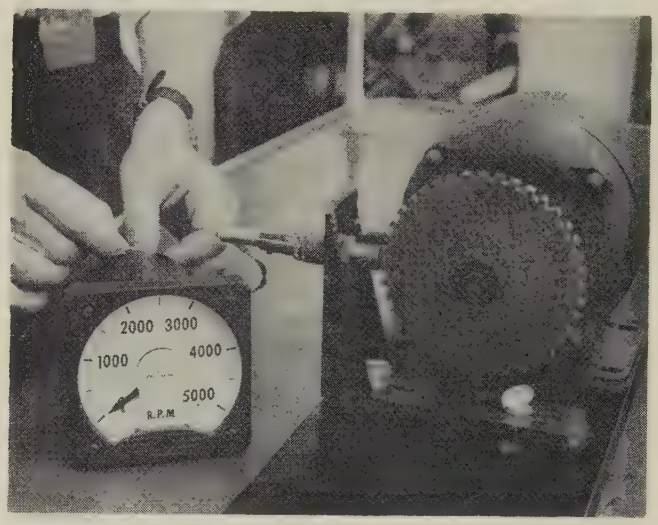
For more information, write Westinghouse Electric Corp. P. O. Box 2099, Pittsburgh 30, Pa.



Forced-ventilated, totally enclosed motor

Speed-Indicator

A new speed-indicator (Type K-241) for use with small steam or gas turbines is now available from the Westinghouse Electric Corp. The principal advantage of the indicating device is that no mechanical connection is required between it and the rotating shaft.



Speed indicator Type K-241

The speed of the rotating part is sensed by an electromagnetic pickup, located near a gear or notched shaft. This signal is applied to an all-static circuit, which transforms the pulses from the pickup to a d-c millivolt signal. The output signal magnitude is proportional to pulse frequency.

For further information, write Westinghouse Electric Corp., P. O. Box 2099, Pittsburgh 30, Pa.

Industrial Fan Series 400

A new line of industrial fans (Series 400) for both material and air handling applications is available from the Westinghouse Electric Corp. A feature of this line is a newly designed open wheel that is versatile and applicable to most material handling problems.

The Series 400 industrial fans can be applied for handling granular, abrasive and fibrous materials, dust collection, fume and high temperature exhaust and similar industrial processes.

For further information, write to Westinghouse Sturtevant Division, Department T-000, 200 Readville St., Hyde Park, Boston 36, Mass.

C & D Batteries

C & D AutoReg® Charger

Newest member of the rapidly expanding line of C & D AutoReg Silicon chargers is an AR-2 model built right into the battery container.

The new AutoReg charges 6-cell, 12-v. motive-power batteries overnight. At present, the charger is built into the container of two different size batteries for trucks with integral battery compartments. One combination consists of the charger with a 6-HC-19 Slyver-Clad® battery whose capacity is 648 amp.-hr. at the 6-hr. rate. The second combination consists of the charger with a 6-CDS-19 Slyver-Clad battery. Capacity of this battery is 540 amp.-hr. at the 6-hr. rate.

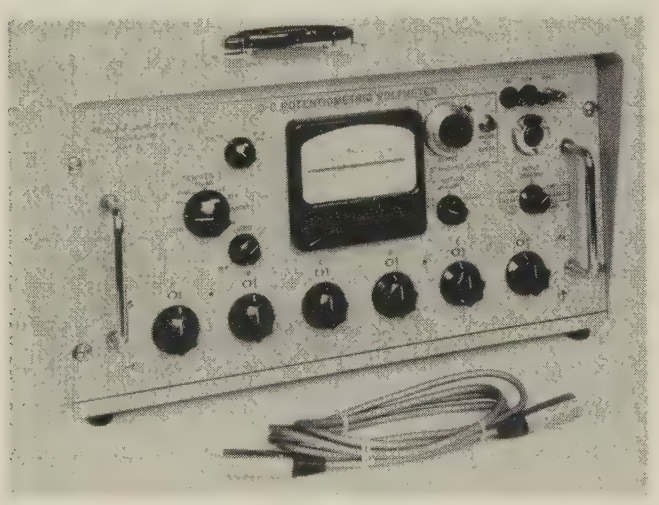
Full details on the new AutoReg charger battery combinations may be obtained by writing the Advertising Department, C & D Batteries, Conshohocken, Pa.

Hallamore Electronics

Portable Precision Voltmeter

A new solid-state precision voltmeter that combines rugged portability with extreme accuracy at high and low ranges is now available from Hallamore Electronics Co., a Division of the Siegler Corp.

The new Hallamore voltmeter, a d.c. potentiometric type, combines an electronic galvanometer with a precision resistor network to enable dial readings down to 0.5 microvolts resolution and up to 10-v. d.c at an accuracy tolerance of $\pm(0.025\% + 3\ \mu v)$. The resistor network is controlled by 6 decade switches covering voltage ranges from $\pm 10\ \mu v$ to ± 10 v.



Portable precision voltmeter

The high precision and the portability of the meter make it especially well suited for both laboratory and production-line measurement work.

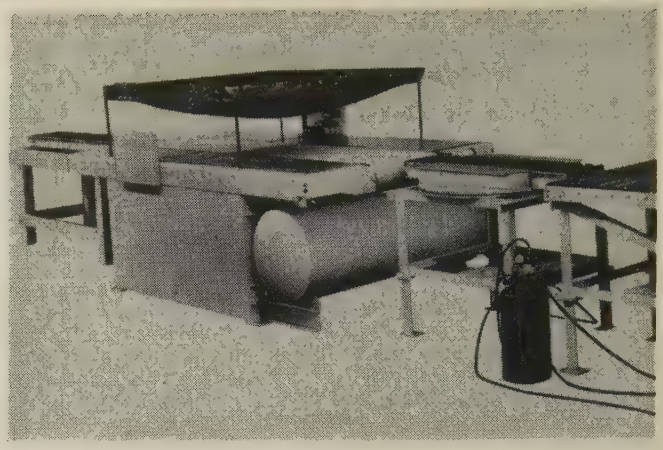
Complete specifications can be obtained by writing to Paul Kemp at the Hallamore Electronics Co., 714 N. Brookhurst St., Anaheim, Calif.

Southworth Machine Co.

Automatic Carton Sealer

The Southworth automatic carton sealer, designed specifically for the paper industry, is an easy to operate, foolproof machine that insures better packaging at lower costs by speeding and improving the sealing of telescopic cartons in which papers are packed.

Operating on a completely automatic schedule, the carton sealer is self-feeding, self-sealing, and self-discharging; and without any adjustments to the machine, will handle any number of different sized cartons simultaneously.



Southworth automatic carton sealer

Used in conjunction with the Southworth sealer is a cold-set adhesive which is quickly applied on all four sides of the cartons with an efficient, production-line applicator. Cartons are then fed automatically onto a vacuum table where they are then automatically vacuum-sealed. An arrangement of electric eyes automatically compensates for varying carton sizes—up to 38 × 50 m.—without the necessity of making any adjustments. A conveyor belt automatically discharges the sealed cartons.

General Electric Co.

General Purpose Control Catalog

A new 72-page publication, GEC-1260D, contains information on the complete line of control devices manufactured by the General Electric Co.'s General Purpose Control Department.

Horsepower selection charts are listed for motors from $^1/_4$ to 200 hp., providing quick selection of starters, heaters, and push button stations necessary for control of these motors.

Appropriate General Electric handbook section and available supplemental bulletins are listed for each product type. General Electric Co., Schenectady 5, N. Y.

Stepless Adjustable Speed Drive

A new, low-cost line of eddy current coupling, adjustable speed drives from 5 to 100 hp. has been announced by the General Electric Co.

The new Kinatrol* line is engineered for a broad range of industrial applications and features simplified design with fewer moving parts for longer service life and sharply reduced maintenance.

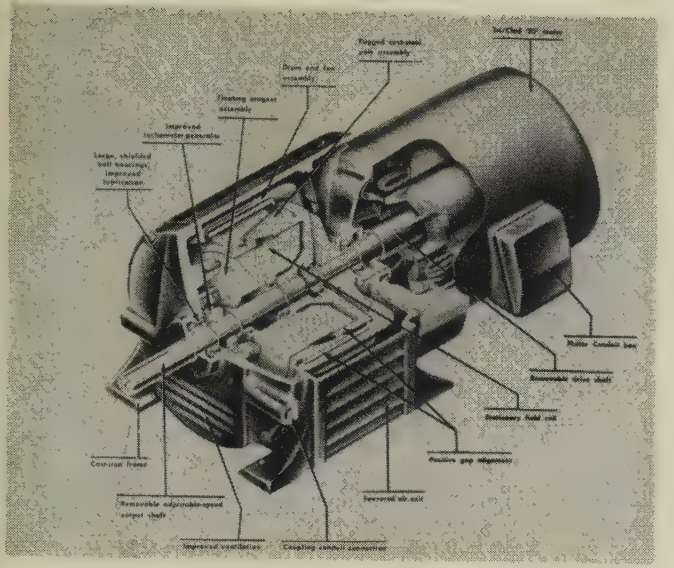
The stepless adjustable speed drive is being offered as a complete package including the drive unit, control enclosure with control for both coupling and integrally mounted Tri-Clad 55* a-c motor, and operator's station.

Versatile performance is offered over a wide speed range. Ratings through 20 hp. are capable of continuous operation down to 100 r.p.m at rated torque providing a speed range of approximately 17:1.

Automatic control provides close speed regulation as standard. On a typical friction type load, for example, regulation of 2% of full speed can be obtained with standard equipment.

Coupling design features include new bearing arrangement assuring positive gap alignment in service and greatly simplified disassembly and reassembly. Bearing loads are sharply reduced for longer bearing life. Generous bearing reservoirs provide continuous controlled lubrication for years of service without attention. Coupling field coil and tachometer generator are stationary, with no brushes or slip rings. Windings are epoxy encapsulated for positive protection. Integral tachometer generator has novel, two-coil, high-fre-

* Trademark of General Electric Co.



Features of the Kinatrol stepless, adjustable speed drives available from the General Electric Co.

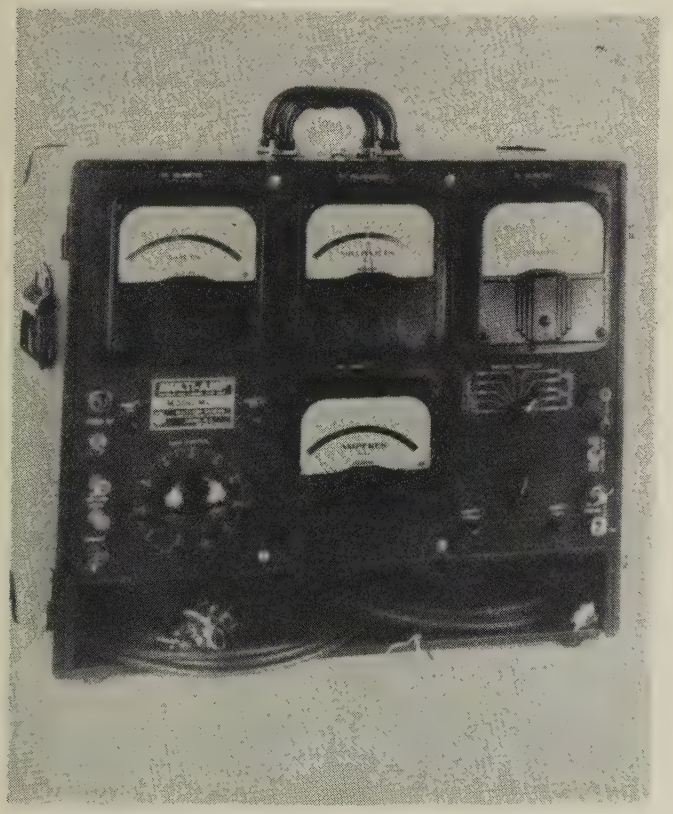
quency design for greater simplicity. Advanced ventilation system gives cooler operation, greater speed range, increased torque capacity, and quieter operation.

Control enclosure elements are all front-wired with exciter-regulator featuring plug-in component board for quick replacement if needed.

For further information, contact the General Electric Co., Direct Current Motor and Generator Department, Erie, Pa.

Jacksonville Service Shop

A new General Electric facility to repair and rebuild all kinds of electrical equipment for industry will open in Jacksonville, Fla. next spring. Target date for having the shop in operation is March 1, 1960.



Indicating instrument calibration set

Multi-Amp Division

Calibrating Set

A portable, compact test unit for calibrating ammeters, voltmeters, and other indicating instruments is one of the late developments of Multi-Amp Division, Multi-Amp Electronic Corp., Union, N. J. It is designed for service in the field, on shipboard, and in the laboratory.

Tracerlab

Scintillation Detector

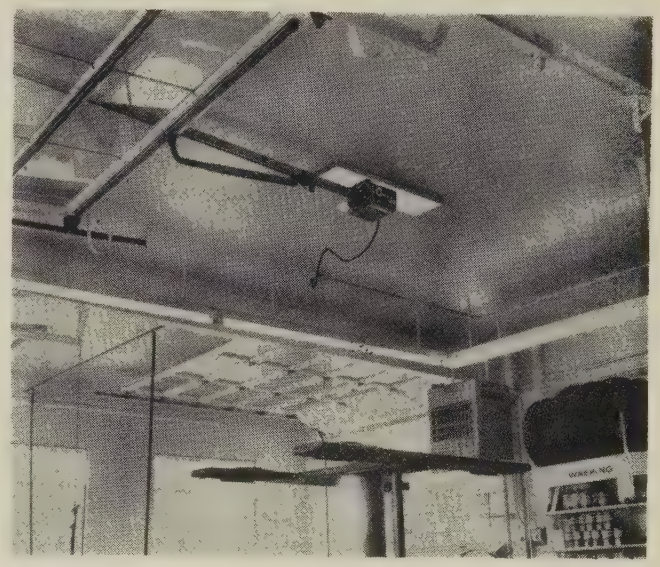
A new, transistorized scintillation detector is now available from Tracerlab. The new unit, the P-20D, has been specifically designed for both clinical and research laboratory applications such as gamma spectrometry, gross gamma assay; and alpha and beta counting. Any one of 11 different detection heads may be used with the main unit.

For further information write to Paul A. McNulty, Public Relations Manager, Waltham, Mass.

The Alliance Manufacturing Co.

Garage Door Operator

A heavy-duty, automatic, overhead door operator especially designed for garage and warehousing needs of the paper products industry was announced by the Alliance Mfg. Co., Alliance, Ohio, a division of Consolidated Electronics Industries Corp.



Alliance heavy-duty, automatic, electronic garage door operator

Called the "Genie Model 410," the unit provides a reliable lifting force of 250 lb., sufficient to operate oversize garage doors up to 10 ft. high and 20 ft. wide. It automatically unlocks, opens or closes, and locks the door at the push of a button, while at the same time turning garage lights on or off.

To simplify operation, the Genie Model 410 employs a single button control. Successive operations of the button cause the door to open, stop, reverse direction, and close.

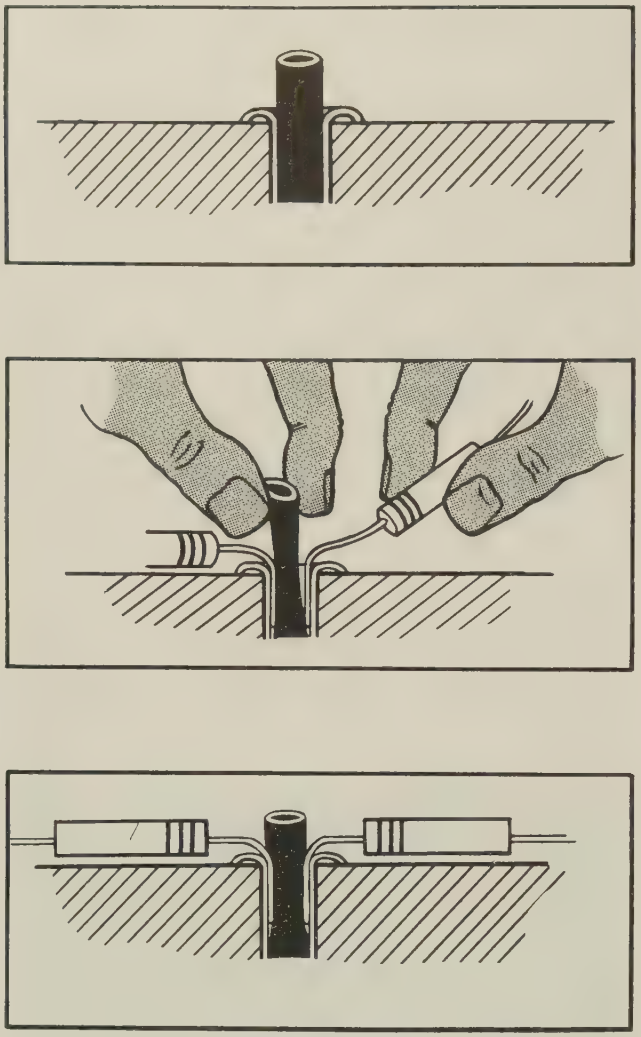
Radio controls for the Genie Model 410 are also available. —A transistorized portable control, the Model *TT-5*.

Plastic Associates

Solderless Connections

A new method of providing fast, solderless connections between wires and component leads has been developed by engineers at Plastic Associates, Laguna Beach, Calif.

The new P/A junction cell consists of a gold-plated eyelet and a long-wearing rubber core (upper drawing). When the



New P/A junction cell by Plastic Associates

core is pulled upward, the rubber stretches, allowing wires to be easily slipped inside the eyelet. When released (lower drawing), the rubber core grips the wires or component leads, pressing them firmly against the gold-plated surface and establishing an electrical contact of essentially zero resistance.

For additional information concerning applications of the new P/A junction cell in electrical and electronic equipment, contact Plastic Associates, 185 Mountain Road, Laguna Beach, Calif.

Appleton Machine Co.

Drum Sheeter

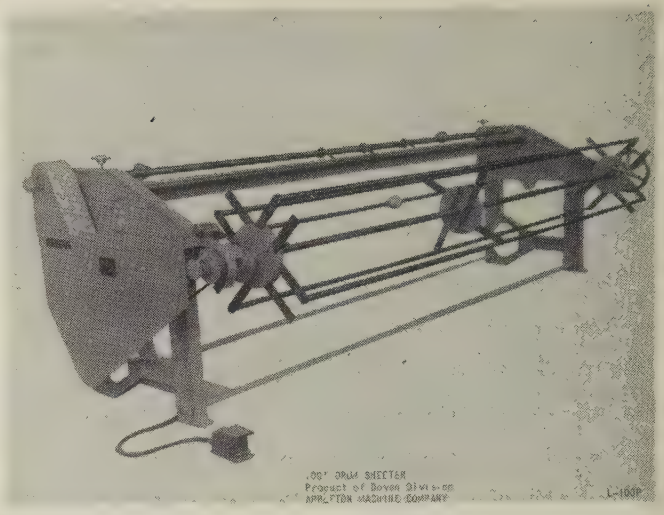
A Drum-type Sheeter to handle 100-in. webs has been announced by the Doven Division of Appleton Machine Co. for use on plastics, polyethylene, acetates paper, cloth, etc. The standard drum can expand to make lengths from 35 to 120 in. and is marked-off in 1-in. graduations. This sheeter can handle over 35 layups per minute. Drum adjustments take less than 10 sec.

The unit is constructed of either cast iron or cast aluminum and is equipped with ball bearings throughout. Optional equipment available include movable foot switches, counters, razor blades for parting material, manual edge guide, and tissue interleave wind-up station.

Centri-Mill

A new cylindrical, stone-filled refiner for use on screen rejects, impregnated chips, and high density pulp has also been introduced by Appleton Machine Co.

The Centri-Mill refines with minimum reduction of fiber



Drum Sheeter by Appleton Machine Co.

length, and has a very low power consumption. Adjustable 3-stage operating clearance of rotor remains constant and unaffected by rate of speed.

Large Gilbert & Nash Air Guide

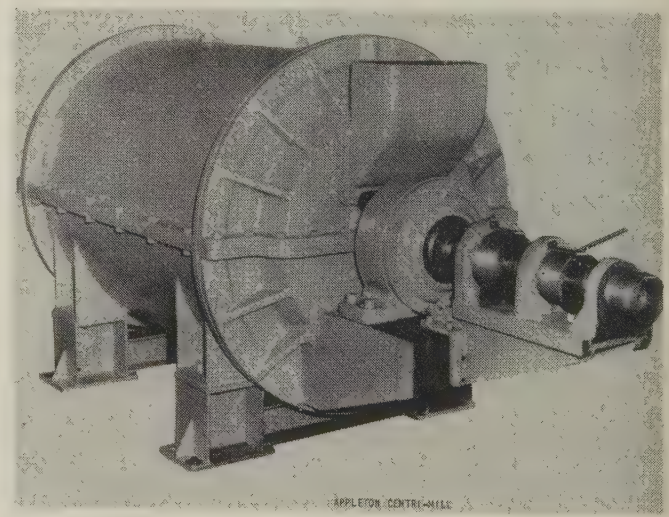
A large size Gilbert & Nash felt and wire air guide is announced by Appleton Machine Co., Appleton, Wis., exclusive manufacturers and distributors of the equipment.

The new air guide (model 560-A) designed for paper machines between 120 and 200 in. wide, is suitable for machine speeds up to 3000 f.p.m. It will fit in any space where a mechanical guide is installed. In most instances, the air guide can be mounted on a paper machine using the existing guide roll, bearings, and backstand.

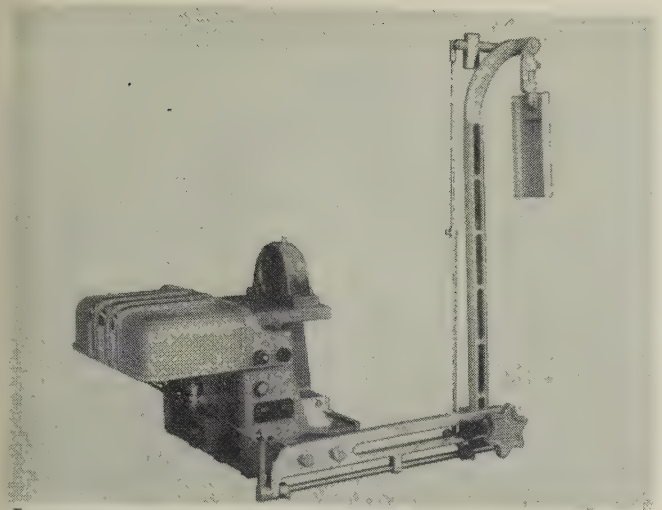
There is no "swing roll" effect or guide roll oscillation because the guide moves laterally in either direction by air pressure and is held securely in place. In the event of air supply failure, the guide roll is held stationary and the wire or felt can be guided manually. There are no springs to pull the guide roll with possible loss of wire or felt.

Standardization of guides on a paper machine is possible because the same model air guide is used for wires, wet felts, and drier felts. Appleton Machine also manufactures the smaller air guide (model 460-A) for paper machines under 120-in. width and mechanical guides.

For more information write: Harry W. Brown, Jr., Manager, Gilbert & Nash Co., Appleton Machine Co., Appleton, Wis



Appleton Centri-Mill



Large size Gilbert and Nash air guide manufactured by Appleton Machine Co.

Dewey and Almy Chemical Division/W. R. Grace & Co.

"Triple Attack" Pulper

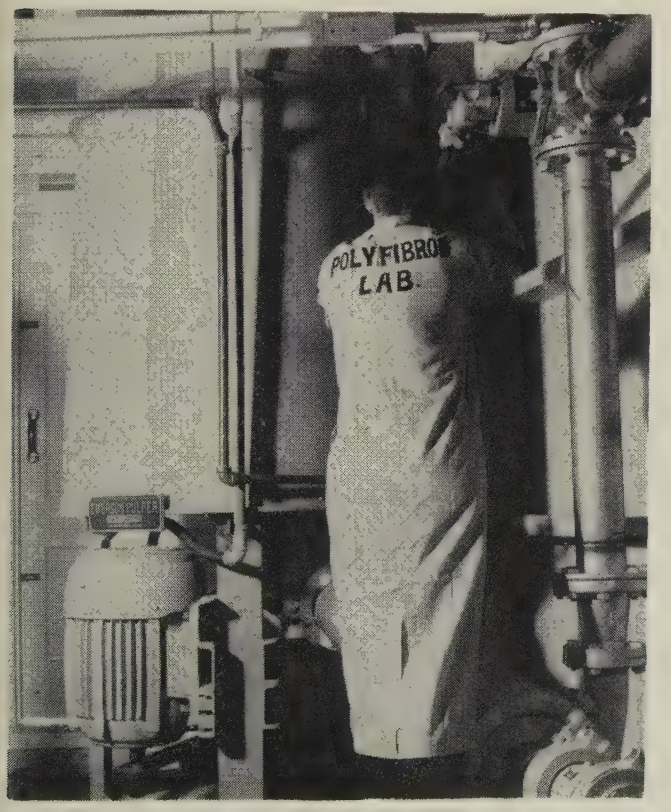
The first laboratory-sized model of the Emerson "Triple Attack" pulper, radical new pulper in which the shape of the tub eliminates the traditional baffles, has been installed in the Polyfibron (polymers-fibers) Laboratory of the Dewey and Almy Chemical Division, W. R. Garce & Co., Cambridge, Mass.

Identical in design to mill-sized pulpers, but with a 100-lb. capacity, the pulper has lobe-shaped, tapering walls. Defibering occurs in three places: at the impellor, along the tub walls, and at a vortex formed where four distinct stock streams, directed inwards and downwards by the lobed and tapered walls, make contact with the faster-moving stock from the impellor blades.

The Dewey and Almy laboratory, where pilot-scale equipment must be capable of processing a wide variety of furnishes, chose the pulper for its pluping efficiency and the stock consistency it will handle—as high as 12%.

Dewey and Almy's model has a vertical-walled extension welded to the top to allow the pulper to serve as a stock chest. Pulp is defibered at high consistency in the lower tapered portion, then diluted and stored in the pulper until used.

The pulper is manufactured by The Emerson Manufacturing Co., Division, John W. Bolton & Sons, Inc., Lawrence, Mass.

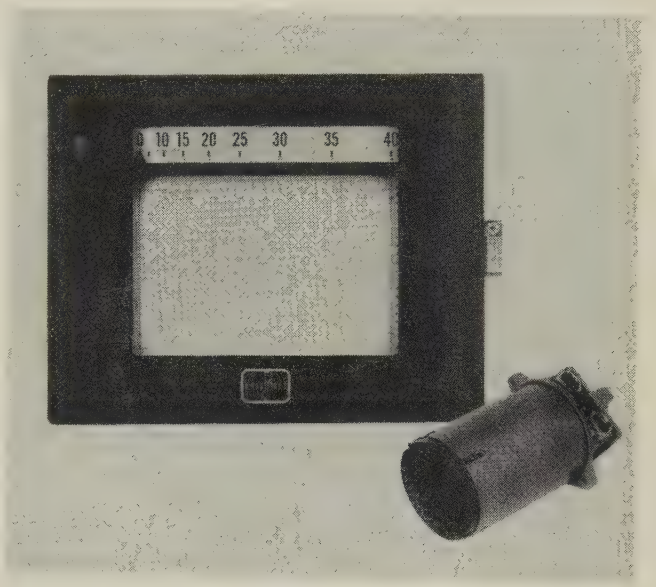


Laboratory-sized model of new Emerson pulper at Dewey and Almy

The Bristol Co.

Pyrometer Measures Temperatures from 0 to 1100°F.

The Bristol Velotron low-range pyrometer system will measure temperatures in ranges from 0°F. to 200°F., up to 1100°F. It is especially applicable for temperature measurement, recording and control, with such products as rubber, synthetic rubber, tiles, roofing shingles, adhesives, cast aluminum, textiles, synthetic and natural fibers, paper, plastics, carpeting, mineral aggregates, chemical flows, petroleum flow, reactor kettles, furnace and oven charges, or exteriors, foundry molds, web-process products, and conveyed, rolled, or extruded products.



The Bristol Velotion low-range pyrometer system

Post Machinery Co.

Arrow Gluer, Series 50-A

Post Machinery Co., Beverly, Mass., have unveiled a new 180° prebreaking straight line gluer which is designated as the Arrow 50-A.

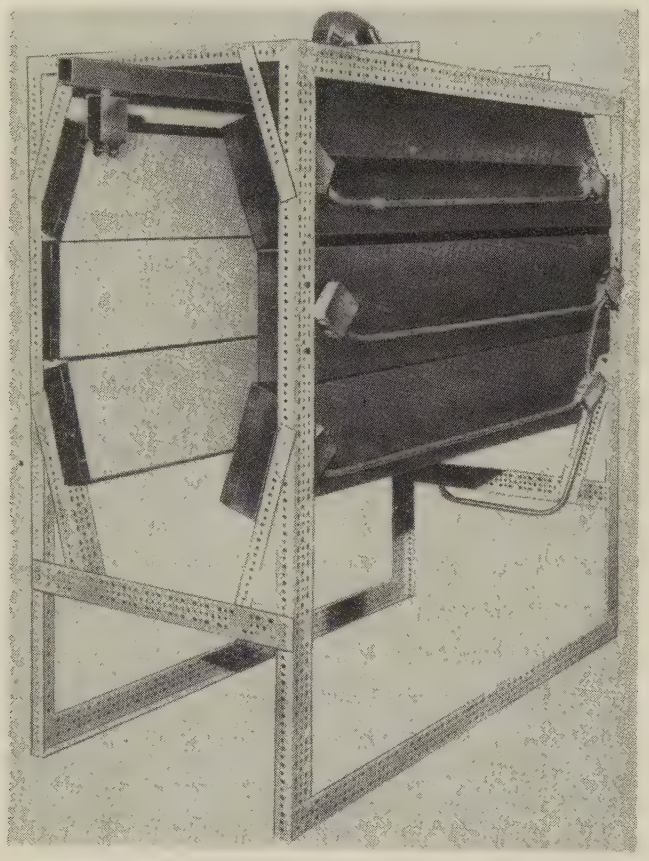
The Arrow 50-A prebreaking straight line gluer by Post Machinery Co.

This gluer will bring to the boxmakers many exclusive features including new power driven "squaring up" nipper rolls, angular yielding flange cage rolls which keep the cartons always under positive control and eliminates belt edge wear, a new heavy duty, variable speed drive for the feed and delivery sections, heavy duty, power driven, dual squeeze rolls, greater accessibility of all controls with complete infinite adjustments, variable width, overhead glue pot wheels and the longest stacker in the industry.

Infra Red Systems Inc.

Heating Ovens

Modular infrared ovens to fitting any drying system and giving uniform heat over entire emitting area are now available from Infra Red Systems, Inc., of Riverdale, N. J. The basic module, RediRay, is a self-contained heater which uses a ceramic face as a source of infrared radiation.



The Redi Ray unit by Infra Red

The advantages of RediRay are several:

1. Uniform, even heat—ceramic face emits heat over *entire* area; no hot spots.

2. Highest efficiency—no bulbs or tubes to get dirty; no reflectors to tarnish or get dirty because *entire* face emits heat.

3. Longer life—no bulbs or tubes to break or burn out; life of protected heating element is indefinite.

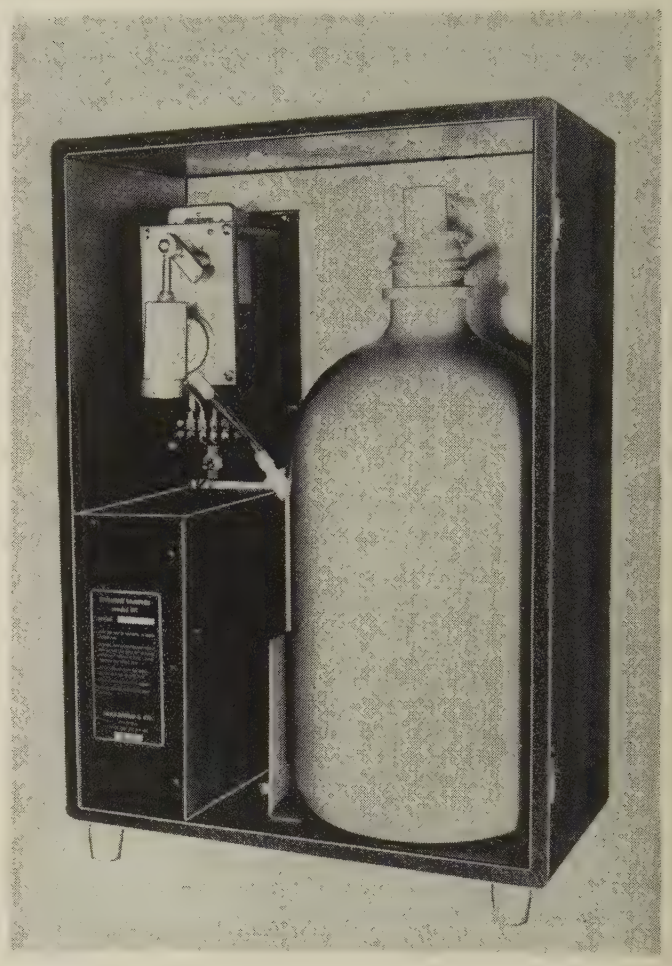
Temperature control is accomplished by any adequate voltage control. The RediRay 6-ft unit shown gives up to 500 w. per sq. ft. radiation. Work temperatures go up to 400°F.

For further information, write: Infra Red Systems, Inc., 240 Route 23, Riverdale, N. J.

Brailsford & Co., Inc.

New Model D.C. Effluent Sampler

Rigid polyethylene components adapt the Model D.C. effluent sampler for handling chemical plant wastes containing a high concentration of organic solvents. The instrument delivers a continuous sample at the rate of 2 gal. per



The Model D. C. effluent sampler

day and is completely independent of either power line or storage batteries. Three no. 6, dry cell batteries will operate the sampler for periods of 2 to 3 months. The valveless pumping mechanism is self-priming and will suction-lift up to 10 it. The pump is driven by a fractional-watt, governed, d.c. motor of patented design with a service life in excess of 2000 hr. All metal parts are corrosionproof and electrical components are independently enclosed. An automatic cut-off is provided to prevent accidental overfilling if the sampler is not serviced on time. The complete unit, in a rugged waterproof plywood case, measures $9 \times 14 \times 20$ in. and weighs 28 lb. including batteries, sample jug, inlet tubing, and strainer.

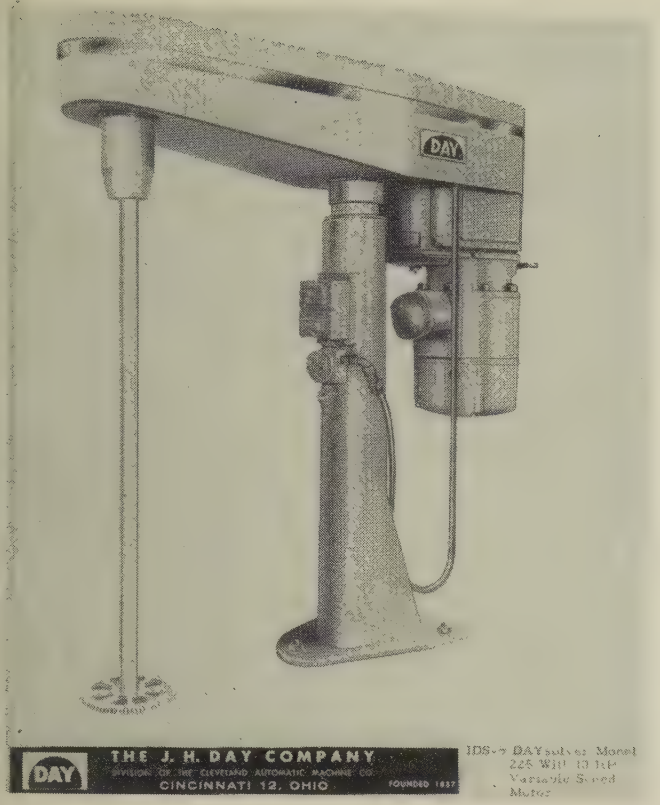
For further information write to Brailsford & Co., Inc., 670 Milton Road, Rye, N. Y.

J. H. Day Co.

Viscous Solution Mixer

The J. H. Day Co., division of The Cleveland Automatic Machine Co., Cincinnati, Ohio, have recently introduced into their line of processing machinery a heavy-duty dissolver for mixing extra-viscous solutions. Known as the "Daysolver," the mixer is built with a heavy-duty, steel column frame and bridge, with oversize stainless steel impeller shaft, to provide smooth, vibrationless operation under extreme work loads.

The bridge swings in a 240° arc and a hoist, operated by 80 lb. of air pressure, provides rapid raising and lowering of the shaft and impeller. A variety of impellers are available, including the newly designed Day "Turbopeller" which com-



The Daysolver viscous-solution mixer

bines five different mixing actions. The "Daysolver" is available in models ranging from laboratory size to a large, 75 hp. unit.

For more detailed information, including specifications, write The J. H. Day Co., 4932 Beech St., Cincinnati 12, Ohio.

Texas Gulf Sulphur Co., Inc.

Manual on Sulfur

Texas Gulf Sulphur Co., Inc., has issued a comprehensive, detailed Sulfur Manual of interest to companies now using or planning to use sulfur, solid or molten.

Topics covered by the Manual are: the sulfur industry, shipping of molten sulfur, handling and storage of molten sulfur, analysis of sulfur, and the physio-chemical properties of sulfur. This part comprises a review of all the properties of sulfur, with the latest proved determinations.

On request, the company will be glad to send the manual and/or individual sections. Write or call Texas Gulf Sulphur Co., Inc., 75 East 45th St., New York 17, N. Y. Attention Sales Department.

Hooker Chemical Corp.

Sodium Chlorate Handling

Outlining "Precautions for the Safe Handling of Sodium Chlorate in Chlorine Dioxide Generation," a new wall chart prepared expressly for tank car users of this chemical in the pulp and paper industry is now available from Hooker Chemical Corp., Niagara Falls, N. Y.

Helpful hints and suggestions based on accumulated mill experience are given for storing and handling sodium chlorate safely and for avoiding fire or explosion hazards. Other topics covered include use of protective clothing and equipment, health hazards, first aid measures, and medical treatment.

Free copies are available to mill men on request. Write to Hooker Chemical Corp., Box 344, Niagara Falls, N. Y.

Employees Education Plan

Hooker Charitable Foundation, Inc., Niagara Falls, N. Y., recently formed by Hooker Chemical Corp., has established a "Corporate Alumnus Program" to encourage financial support by its employees of universities, colleges, their graduate schools, and accredited 2-year junior colleges and technical institutes.

In making the announcement, Thomas E. Moffitt, Foundation president who is also president and chief executive officer of Hooker Chemical, said that this "gift-matching plan will become operative December 1."

The program provides essentially that the Foundation will contribute an amount up to $500 per employee in any fiscal year (December 1 through November 30) equal to the sums given by an employee of Hooker Chemical, or its subsidiaries, to or for the benefit of a qualifying institution of higher education. It makes no difference whether or not the employee attended that institution.

The Foundation will direct and administer the Corporate Alumnus Program as well as handle certain other types of corporate giving. Other Foundation officers, all Hooker Chemical officials, are: R. Wolcott Hooker, vice-president; Thomas F. Willers, treasurer, and Ansley Wilcox 2d, secretary.

Appointments

Joseph E. Thornberg, 1503 Cleveland Ave., Niagara Falls, N. Y., has been appointed to the newly created position of manager-market research, in the sales department of the Eastern Chemical Division, Hooker Chemical Corp.

The Eastern Chemical Division, with headquarters at Niagara Falls, has sales offices in Buffalo, Chicago, Detroit, New York, Philadelphia, and Worcester, Mass. Manufacturing plants of the Division are located in Niagara Falls, Montague, Mich., and Columbus, Miss.

Union Carbide Chemicals Co.

Physical Properties Booklet

A new 28-page Physical Properties booklet, describing more than 400 synthetic organic chemicals, has been published by Union Carbide Chemicals Co.

The booklet contains information on applications, physical properties, and shipping data. Included are alcohols; acids; anhydrides; chlorine compounds; esters; ethers and oxides; glycol-ethers; glycols and triols; ketones; monomers; Carbowax polyethylene glycols; metallic salts; nitrogen compounds; cyclic compounds; Flexol plasticizers; polypropylene glycols; Ucon fluids, lubricants, and fluorocarbons; Tergitol surfactants; Polyox water-soluble resins; Cellosize hydroxyethyl cellulose; new chemicals for evaluation; and chemicals for special applications.

Hydroxyethyl Cellulose Booklet

A new 16-page booklet, describing the properties and uses of Cellosize hydroxyethyl cellulose, has been published also by Union Carbide Chemicals.

The booklet contains extensive data on properties, preparation of solutions, film and coating properties, preparation of water-insoluble films, applications, toxicological properties, specification limits, shipping data, and test methods.

Cellosize hydroxyethyl cellulose is a nonionic synthetic colloid that is readily soluble in hot or cold water. It has excellent tolerance for dissolved electrolytes. It can be used as a thickener, binder, stabilizer, protective colloid, film-former, or coating.

National Carbon Co.

Heat Exchangers and Steam Jets

A new 12-page illustrated bulletin on Karbate impervious graphite immersion heat exchangers and circulating steam jets for heating or cooling corrosive solutions in all types of

tanks is available from National Carbon Co., Division of Union Carbide Corp.

Separate sections on plate, bayonet, and coil type immersion heat exchangers include general descriptive, installation, and operation information. Dimensioned sketches of each type give complete construction details, and tables present information on weights and effective surface areas. Specifications on circulating steam jets include dimensions and installation instructions.

Nomograms and curves present design information, and sample calculations illustrate the proper application of the equipment under a wide range of operating conditions.

For a copy, write for Catalog Section S-6620 to National Carbon Co., 535 Fifth Ave., New York 17, N. Y.

Karbate Entrainment Separators

Entrainment separator modules made of impervious graphite for effective separation of entrained liquids from corrosive gas streams are now available from National Carbon Co., Division of Union Carbide Corp. Designed for high collection efficiency and low pressure drop, the corrosion-resistant modules measure 1 by 2 ft. and can be easily assembled into banks to fit any duct or process vessel.

Type MV entrainment separator modules are of unitized construction, and operate on the principle of impingement plus venturi action. Staggered rows of tear-drop-shaped Karbate impervious graphite rods are cemented in place with a minimum of $^1/_4$-in. clearance to minimize clogging. The staggered arrangement prevents straight-through flow, and the open diagonal lanes permit easy cleaning when necessary. The streamlined rod shape retards re-entrainment and minimizes resistance to gas flow.

Modules are available in two styles: style A is used in single-row entrainment separators or as the top row of larger assemblies; style B units for the lower rows have a gutter and downcomer to provide positive drainage of the liquid separated from the gas by upper units. Side plates for both styles provide proper fit to the wall of the containing vessel.

For more information on modular, type MV, entrainment separators write National Carbon Co., 535 Fifth Ave., New York 17, N. Y.

Antara Chemicals

PVP/VA Copolymers

An expanded and comprehensive bulletin on new types of commercially available vinylpyrrolidone/vinyl acetate copolymers and their numerous applications has just been issued by Antara Chemicals, sales division of General Aniline & Film Corp. These materials were first introduced as 50% ethanol solutions. They are now available in various physical forms as well as in several monomer ratios. Solid, emulsion, and solution types now provide specific materials suitable for diverse processing conditions and product requirements, and for use in aqueous as well as organic solvent or aerosol systems.

A copy of Bulletin AP-88 Rev. is available by writing to Antara Chemicals, General Aniline & Film Corp., 435 Hudson St., New York 14, N. Y.

Solvay Process Division/Allied Chemical Corp.

Hydrogen Peroxide

A new technical bulletin on hydrogen peroxide has been published by the Solvay Process Division of Allied Chemical Corp. The 63-page booklet presents practical information on the properties of hydrogen peroxide and its aqueous solutions, and on the handling, dilution, and storage of the product.

Stickle Steam Specialties Co.

Condensate Flow Regulator

Bulletin 300-M shows a new type condensate flow regulator said to do a better job at less cost than steam traps.

Designated as Series 300 micro adjustable orifice, the regulator with glass case is designed for drainage of heating and processing units operating continuously at pressures up to 60 p.s.i. Models with a metal case handle pressures up to 200 p.s.i.

It is adjustable, operates without moving parts, and provides a constant visual check on the flow of condensate and temperature of steam inside the unit. Scale and other impurities which clog a steam trap, can easily be flushed through the orifice without harm.

Tables furnished with the bulletin give the capacity of condensate handled under different pressure differentials and other conditions.

The bulletin and tables are available from the manufacturer, Stickle Steam Specialties Co., 2215 Valley Ave., Indianapolis 18, Ind.

The Borden Chemical Co.

Nontoxic Adhesive

The availability of a new list of approved nontoxic adhesives for food packaging applications has been announced by The Borden Chemical Co. The list includes a series of resin based emulsion glues specially designed for convolute and spiral-wound food containers such as used for hot and cold beverages, meats, condiments, frozen foods, dairy products, bakery goods, drinking straws, folding cartons, and paper laminating.

Food clearance data from independent testing laboratories are available upon request. All Borden nontoxic adhesives are provided in a wide range of setting speeds in ready-to-use form. Five-gallon trial quantities are also being offered. For further information write Department H, The Borden Chemical Co., 350 Madison Ave., New York 17, N. Y.

Expansion

Completion of a new pilot plant, first step in the $\$2^1/_2$ million expansion program at its Leominster, Mass., operation, has been announced by the Borden Chemical Co.

Designed to produce up to 1000-lb. lots of polyvinyl alcohol, polyvinyl acetate, acrylics, butadiene-styrene and other products, the pilot plant is being operated by Borden Chemical's Polyco-Monomer department. It was designed and planned by the company's central engineering department and built by United Engineers and Constructors.

Currently under construction at the Leominster site are new development and technical service laboratories, a new unit for the production of butadiene-styrene products, a new polyvinyl alcohol plant using a Borden-designed continuous process, and facilities for the manufacture of Lemoflex, a special resin used in producing water-soluble packaging film.

C. W. Smith, general manager of the Polyco-Monomer department, said that all the new facilities are expected to be completed and in operation by early next year.

Appointments

Maurice S. Letourneau, New York District sales manager for the Borden Chemical Co.'s Polyco-Monomer Department since last August, has been promoted to Eastern District sales manager, it was announced by W. G. West, department sales manager.

In his new position, Mr. Letourneau will be responsible for directing the sale of Borden's full line of polymers for the textile, paper, paint and adhesives industries in the New England, New York, Philadelphia, and Southern market areas.

Sprout, Waldron & Co., Inc.

Pulp in Pellets

The availability of an up-to-date technical article on the subject of pelleted pulp has been announced by Sprout, Waldron & Co., Inc.

The article contains data on a variety of pulp pellet test runs involving groundwood, kraft, unbleached sulfite, bleached semichemical and bleached kraft. Basic information on three pilot plant installations is also included.

For your free copy, ask for *Better Fibers*, 1, No. 5, or for the latest bulletin on pulp pellets.

Testing Machines, Inc.

Basis Weight Scales

A data sheet on the various types of quadrant basis weight scales in now available from Testing Machines, Inc. The scales illustrated and described are of the pendulum type with jewel or hardest steel bearings. Models include Standard Paper Desk Scales, Pocket Paper Scales, Bundle Scales and Special Purpose Scales.

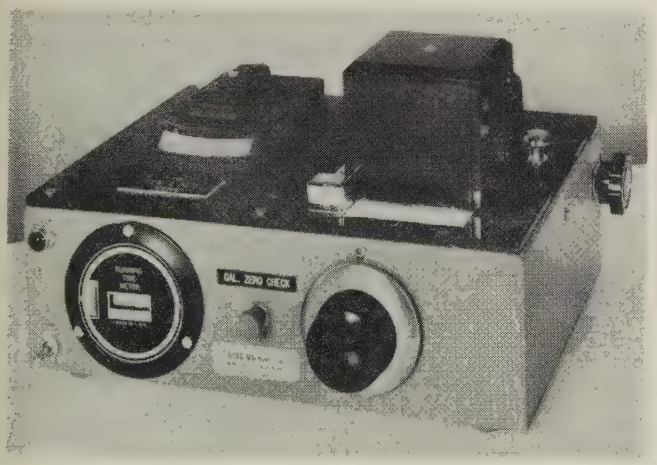
Friction & Slip Tester

Testing Machines, Inc. has available a 2-page brochure which illustrates the new TMI Friction & Slip Tester. The brochure describes the processes involved for testing the friction and slip characteristics and qualities of paper, paper board, and corrugated medium.

Pen-A-Size Tester

The new, automatic, modernized, photoelectric, penetration, and sizing tester for paper accurately and automatically measures the absorption of a colored liquid (usually a standard ink) into the test specimen.

The TMI Pen-A-Size tester uses a well-proved, patented system of balanced photoelectric cells in combination with a filter screen of known value.



TMI's photoelectric penetration and sizing tester for paper

Twist a test knob and ink is applied to the under surface of the specimen as automatic timing begins.

A digital read-out timer with $^1/_{10}$-sec. sensitivity automatically stops when penetration of the liquid ink has reduced reflectance of the upper surface of the test specimen to an amount equal to the calibrated screen.

Bailey Meter Co.

Multipointer Gage Unit

Multipointer gage units, used for pressure and level measurements, and pneumatic transmitting or receiving, are described and illustrated in a new, 4-page Product Specification M42-2. Actual size drawing of 7 in. indicating scale is included to illustrate easy readability. Literature available on request from manufacturer, Bailey Meter Co., 1050 Ivanhoe Road, Cleveland 10, Ohio.

Minneapolis-Honeywell Regulator Co.

Gas Chromatograph

Instrumentation Data Sheet 10.15-20 describes how the F & M Model 202 Gas Chromatograph, employing an ElectroniK recorder, has increased the speed, scope, and accuracy of gas chromatographic analysis. The sheet also describes the linear programmed temperature method of operation, and shows chromatograms made using this method as compared to chromatograms made using a constant temperature.

Thermocouples

Specification sheet No. SOOl-1a contains a comprehensive explanation of the subject material based on the latest data from ASME. Graphs are also included to help select the proper well design and material. For your copy send a request to Minneapolis-Honeywell Regulator Co., Wayne & Windrim Aves., Phila. 44, Pa.

MV/P Transmitter

Honeywell's new ElectriK Tel-O-Set MV/P (millivolt-to-pressure) transmitter measures d.-c. millivolts and resistance inputs (thermocouple, pH preamplifiers, resistance thermometers, etc.) and transmits a standard 3 to 15 p.s.i. pneumatic signal. Write for Specification FS 1003-2a for details, ordering information, typical applications, method of operation, and features.

Associated Control Equipment, Inc.

Servo-Gauge Metering Pump

Complete details on the Servo-Gauge metering pump are provided in a bulletin now available from Associated Control Equipment, Inc., P. O. Box 136, Coraopolis, Pa.

The bulletin describes operating principles of the pump and its controller, which has a pump-plunger-positioning accuracy within 0.002 in., repeatedly. Also included are five typical applications which are fully explained in terms of suggestions for similar uses. Design data listed include maximum capacity, operating characteristics, stroking rate, materials of construction, and actuating power requirements.

The bulletin, No. P-21, is available upon request to Associated Control Equipment, Inc., P. O. Box 136, Coraopolis, Pa.

B-I-F Industries, Inc.

Proportioning Pump for Corrosive Chemicals

A new, lost cost, electrically-driven Chem-O-Feeder for the accurate proportioning of Concentrated Corrosive Chemicals has been announced by B-I-F Industries, Inc., manufacturers of process instrumentation, equipment and systems for positive control of materials in motion.

The diaphragm-type pump, Model 1331 Proportioneers Chem-O-Feeder, is made of corrosion-resistant materials and will effectively meter such chemicals as 66° Bé., sulfuric acid, sodium hydroxide, phosphoric acid, photographic solutions, hydrofluosilicic acid, etc. Featuring adjustable stroke length while in motion, the pump is ideal for the chemical and air conditioning fields and general industry.

Lunkenheimer Co.

Valve Catalog

A newly revised version of its comprehensive 500-page valve catalog, "Catalog '60" is announced by the Lunkenheimer Co., Cincinnati, Ohio, manufacturers of quality industrial valves and lubricating devices. The most complete

and comprehensive catalog of its type, the new Lunkenheimer publication features a special 24-page valve selector and over 100 pages of valuable engineering data. In addition, the complete line of steel, iron, bronze, and PVC valves is presented, along with specifications and illustrations on lubricating devices, boiler mountings, cocks, and other products. Chief new items since the last printing of the Lundenheimer catalog are complete specifications on LQ600 Bronze Globe valves, Luncor PVC valves, and Lunkenheimer forged steel gate valves.

Copies of the new catalog are being distributed personally by authorized Lunkenheimer distributors and sales representatives.

Catalog Guide for Process Industries

A new catalog guide of equipment for the process industries has been released by Allis-Chalmers.

Included in the guide are descriptions of compressors, vacuum, centrifugal, axial flow and turbine pumps, and valves for fluid flow; crushers and grinding and roller mills for disintegration, mixing and agglomeration; vibrating and gyratory screens for dry separation; blade mills, pool washing screens and log washers for liquid separation; kilns, driers, slakers, pelletizers, coolers, and liquid heaters for heat transfer.

The guide also covers electrical equipment for the complete process plant, electronic metal detectors, water conditioning equipment, chemicals and service, as well as car shakers, lift trucks, tractors, etc.

Copies of "Allis-Chalmers Equipment for the Process Industries," 25C6177P, are available on request from Allis-Chalmers, Milwaukee 1, Wis.

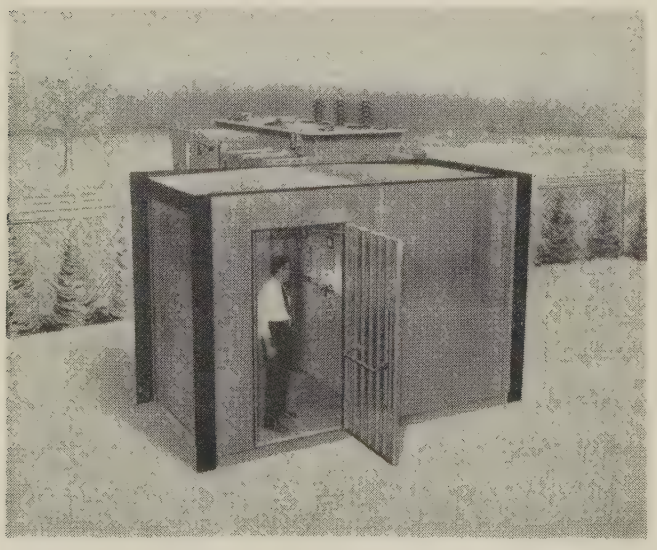
Allis-Chalmers Manufacturing Co.

All-Aluminum Outdoor Switchgear Housing

One answer to outdoor switchgear housing corrosion problems was introduced by Allis-Chalmers. The entire housing for d.-c. switchgear is made of extruded aluminum panels, developed in conjunction with the Reynolds Metals Co. Aluminum extrusions are snapped together to form a maintenance-free structure for outdoor use.

Called the Aluma-Clad switchgear housing, the new type structure requires no paint to protect it from weather wear. Aluminum surfaces do away with the need for primers, enamels, sealers, and also the task of periodic repaintings.

The new Aluma-Clad housing design permits an increased flow of air through filters in the bottom and out of the top of the housing. This feature is a result of the open-labyrinth



The all-aluminum housing for outdoor switchgear

Panels for the Aluma-Clad switchgear housing

cross section of the panels which snap together to form walls, floor, and roof. Major sections are joined by aluminum alloy bolts with closed-cell, neoprene gasketing between adjacent panel sections.

Directed air flow within the switchgear housing keeps internal heat at a minimum. Heating from external radiation is negligible due to the high degree of reflectivity of the outside surfaces.

Appointments

Donald H. Kuenning and Travis L. Lindsay have been assigned as sales representatives to the Milwaukee and Jacksonville (Fla.) districts, respectively, of Allis-Chalmers Industries Group.

Corning Glass Works

Printed Circuits

Electronic designers can now make, and remake, their own high quality printed circuit layouts—without leaving their labs—with a new product made by Corning electronic components.

The Fotoceram grid board is clad on both sides with copper that can be etched away as desired. The grid for installation of components consists of 0.052-in. roundholes spaced $^1/_{10}$-in. apart on center.

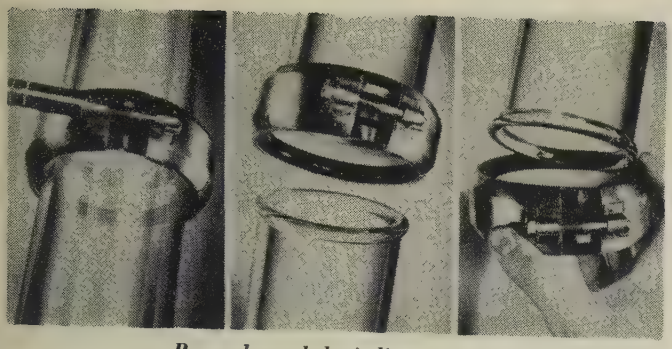
Fotoceram is a glass-ceramic developed by Corning. It has high strength, high temperature resistance, zero water absorption, and is nonflammable and dimensionally stable.

Detailed specifications on the grid board are the same as those published in Corning's Bulletin CD9.01 for printed circuits made to customer design. The bulletin is available from the Electronic Components Department, Corning Glass Works, Bradford, Pa.

Pyrex Brand Drainline

A 12-page illustrated bulletin on the new Pyrex brand lifetime drainline system for disposal of corrosive wastes has been issued by Corning Glass Works.

The system of corrosion-resistant, tempered glass piping and fittings features a one-piece coupling designed to make a quick, permanent compression joint. The drainline has been engineered for vertical or horizontal mounting and for burial in the ground.



Pyrex brand drainline system

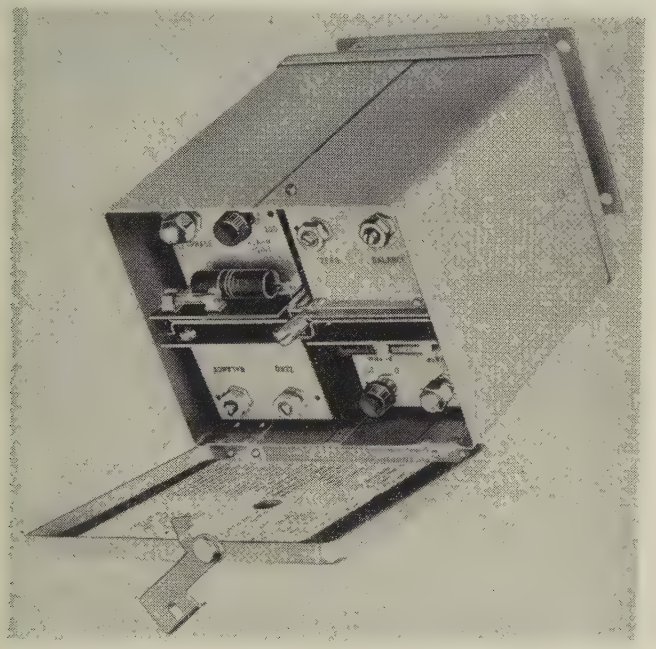
The bulletin contains product and property data, information on making joints and hanging, and a complete list of available fittings. Schematic drawings and tables are included.

The bulletin, PE-30, is available on request from the Plant Equipment Sales Department, Corning Glass Works, Corning, N. Y.

Swartwout Co.

Electronic Alarm

A new, self-powered electronic alarm, the R24, can actuate an annunciator or perform an auxiliary control function when a process signal reaches a preset point. Introduced by the Swartwout Co. of Cleveland, Ohio, the alarm is equipped with a built-in power supply and adjustable alarm-point signal and the unit can be installed with the alarm-point set from a remote location.



Swartwout electronic alarm R2A

Additional information is contained in Specification Sheet A809, available from the Swartwout Co., 18511 Euclid Ave., Cleveland 12, Ohio.

The Carpenter Steel Co., Alloy Tube Division

Corrosion Resistant Tubing

A new bulletin on tubing and pipe made of Hastelloy Alloys B and C has been issued by The Carpenter Steel Co.'s Alloy Tube Division, Union, N. J.

The bulletin outlines applications of these super corrosion resistant alloys and describes their resistance to corrosive fluids.

For further information or to obtain a copy of this 6-page bulletin, write to the company.

Whiting Corp.

Material Handling Booklet

A 16-page booklet entitled "Plan Ahead with Whiting Material Handling Equipment" is being offered by Whiting Corp., Harvey, Ill. The booklet describes the wide variety of material handling devices offered by Whiting. Overhead traveling cranes, trambeam monorail and crane systems, pressuregrip vacuum handling systems, and trackmobile, the road and rail freight car mover, are illustrated and described.

I-T-E Circuit Breaker Co.

Molded Case Circuit Breakers

The electrical industry's most complete and diversified line of molded case circuit breakers is described in a comprehensive new bulletin published by I-T-E Circuit Breaker Co., Philadelphia, Pa.

The illustrated, 40-page, 2-color bulletin gives ratings, specifications and operating characteristics as a guide to designers, contractors and consulting engineers in selecting breakers for a wide variety of applications.

Copies of the bulletin, No. 5001-1A, may be obtained from I-T-E Circuit Breaker Co., 1900 Hamilton St., Philadelphia 30, Pa.

General

Oyster Survival

The Northwest Pulp and Paper Association has produced a 26-min. color motion picture on oyster survival which is available for showing now.

Called "Oyster Development and Survival," the film gives an intimate look at life in the waters of the Pacific Northwest. It probes many of the baffling mysteries of what affects the life of oysters and traces oyster life from the beginning—as eggs are being fertilized—to full development.

The camera takes viewers inside marine laboratories of industry, universities, and government to show how marine biologists are working to solve the many problems that exist in an area where both oyster culture and other industries are important.

The film will appeal to everyone interested in science, the field of marine biology, and particularly the techniques and problems of oyster culture. It is available through Northwest Pulp and Paper Association, 202 Tacoma Building, Tacoma 2, Wash.

U. S. Forest Products

Reports Available

Two new reports relating to sandwich construction and design are among the seven new or revised reports recently issued by the U. S. Forest Products Laboratory, Madison, Wis.

"Structural Sandwich Design Criteria," Report 2161, presents basic design philosophy for structural sandwich construction under various types of loading. Report 2165, "Long-Term Case Study of Sandwich Panel Construction in FPL Experimental Unit," describes the 12-year exposure and service data that was compiled on sandwich panel construction in an experimental house unit at the Forest Products Laboratory.

"Creep of Small Wood Beams Under Constant Bending Load," Report 2150, is an account of a series of experiments conducted at the Forest Product Laboratory to determine the behavior of small, clear, simply supported Douglas-fir beams in controlled atmospheres under constant dead

loads of predetermined size that were applied until failure of the beam occurred.

"The Woods of Liberia," Report 2159, is a 157-page illustrated publication containing pertinent data on approximately one third of the trees known to grow in Liberia.

Machines suitable for the production of chips to meet various specifications of new products utilizing wood chips are described in Report 2160, "Wood-Chipping Equipment and Materials Handling."

"Preservatives for Wood Pallets," Report 2166, points out various preservatives and methods of application that may be used advantageously for lengthening the service life of wood pallets.

Information on an 8-ft., tunnel-furnace test used to investigate surface flammability for building materials has been incorporated along with other pertinent data in a recently revised publication entitled, "Fire-Test Methods Used in Research at the Forest Products Laboratory," Report 1443.

In addition to the reports, the Laboratory has issued revisions of "Partial List of Publications for Furniture Manufacturers, Woodworkers, and Teachers of Wood Shop Practice," Report 1775 and "List of Dry-Kiln Companies and Engineers and Consultants in the United States," Report 1031.

Single copies of these reports are available free upon request from the Director, Forest Products Laboratory, Madison 5, Wis.

At Home with Wood

The continuing scientific study of wood utilization carried on at the Forest Products Laboratory is featured in a new film, "At Home with Wood," recently produced in color by the Forest Service, U. S. Department of Agriculture.

The film shows how research work conducted at the Laboratory leads to new and improved uses for wood and wood products found in American homes. It tells the layman about technicians at the laboratory who literally tear apart various species of wood to find its most efficient and economical use.

New types of home constuction, including stressed skin panels and structural sandwich construction of houses, are explained in the film. It also shows research going on with wood preservatives, paints, and outside finishes. In addition, the film points out that such common items as the plastic wrap, and hamburger wrapping are representative of wood products found in everyday life.

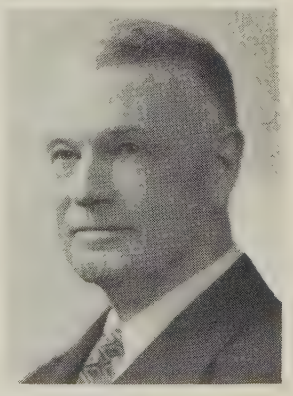
"At Home with Wood" is available to all interested groups on a free rental basis merely by contacting the Regional Forester, at the Forest Service regional office nearest you, or the Director, Forest Products Laboratory, Madison 5, Wis.

American Forest Products Industries

Appointments

John H. Hinman, chairman of the board of International Paper Co., N. Y., was elected 1960 president of American Forest Products Industries, sponsor of the nationwide Tree Farm program of growing trees as a crop on private lands.

W. R. Adams, St. Regis Paper Co., New York, was elected a vice-president, J. D. Bronson, Cascade Lumber Co., Yakima, Wash., was also re-elected a vice-president, and D. B. Frampton of D. B. Frampton & Co., Columbus, Ohio, was re-elected treasurer. Charles A. Gillett of Washington, D. C., was renamed managing director.



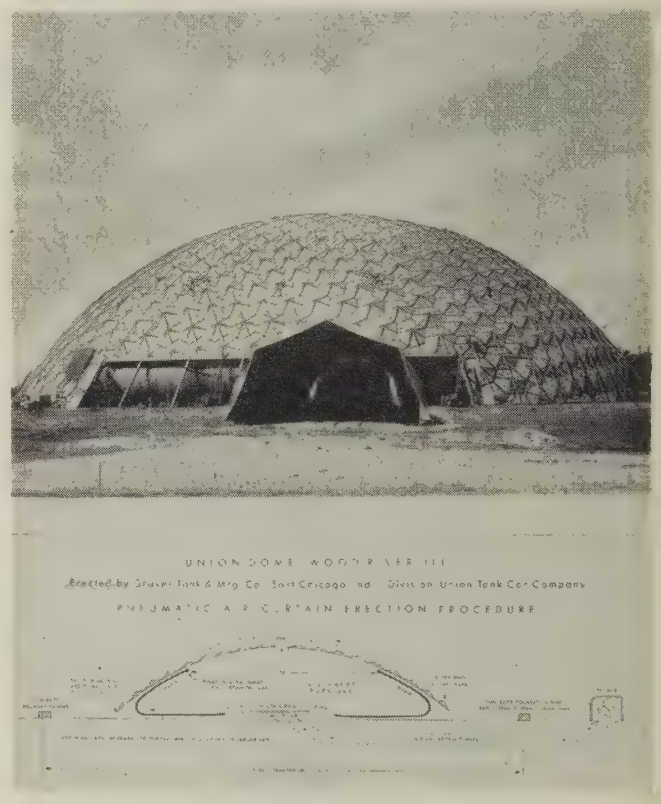
John H. Hinman

Representatives of forest industries throughout the nation heard reports that the 19-year-old voluntary Tree Farm movement had passed the 50-million acre mark in accredited Tree Farms.

Wood River Dome

Graver Tank & Mfg., Co., a division of Union Tank Car Co., has completed the largest structure in the world ever to be built from the top down.

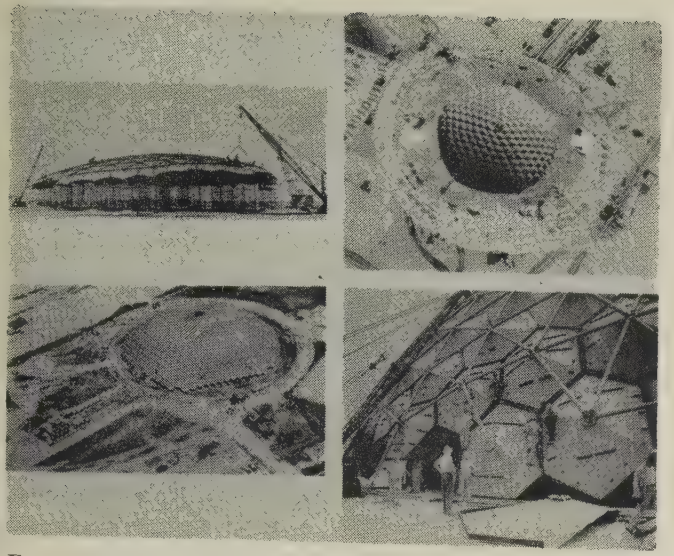
The giant circular building is the Union Dome in Wood River, Ill., 25 miles north of St. Louis. Constructed of nearly 1000 hexagonal steel panels welded together, the large dome-shaped structure is comparable in height to a 10-story office building and large enough inside (100,000 sq. ft.) to hold a baseball or football field.



The Wood River Dome was designed, fabricated, and erected by Graver for use by Union Tank Car Co. of Chicago as a repair and maintenance facility for railroad tank cars. The entire project, including equipment to go inside the dome, is scheduled for completion in mid-1960.

In erecting the dome by starting at the top, Graver employed a huge, inflatable, nylon fabric bag with which it literally lifted the dome on air. The entire dome shell—560 tons of steel, 120 ft. in height and 380 ft. in diameter at the base—rested on 1.6 oz. per sq. in. of air pressure. It was kept from "floating" on the bag by resting 10% of its weight on a circle by hydraulic jacks and 10 guy lines attached to the dome. The unusual operation made it possible to do practically all construction work at or near the ground. As work progressed, the dome was raised pneumatically to successively higher levels, making room for the addition of another row of panels at ground level.

Technically known as a pneumatic lifting diaphragm, the vinyl coated nylon bag—$^1/_{32}$ in. thick—was made to Graver's specifications by Domestic Film Products Corp. of Millersburg, Ohio. Although pneumatic erection operations of a similar nature have been carried out in the past, this was the



Four stages in the construction of the Union Dome at Wood River, Ill.

largest diaphragm and the biggest top-to-bottom construction project on record.

In size, the Wood River Dome and its sister dome at Baton Rouge, La., completed last year, are the world's largest circular buildings without internal or external supports. The Baton Rouge Dome, which also serves Union Tank Car Co. as a repair and maintenance facility for tank cars, was constructed along conventional lines, beginning at the foundation and working up. Certain other design changes were made at Wood River which, along with the pneumatic erection process, are expected to result in a cost lower than the approximately $10 per sq. ft. cost of the Baton Rouge Dome shell.

Among potential uses for big dome structures are sports arenas, civic auditoriums, factories, shopping centers, hospitals, industrial buildings and any structure which is utilized for mass assembly of workers or spectators.

The Folding Paper Box Association of America

Annual Meeting

The Folding Paper Association of America will conduct its annual meeting March 22 and 23 at the Ambassador Hotel, Los Angeles. The traditional public showing of its annual folding carton competition will be the following day at the Ambassador. All entries will then go to Chicago, where they will be on public display March 30 and 31 at the Drake Hotel. On April 12, 13, and 14 they will be exhibited at the Coliseum in New York.

The Annual Report

Research Bureaus, Inc., announces publication of the volume entitled, "The Preparation of The Annual Report—a Document of Modern Business."

The information contained in the volume embraces all phases of the preparation and production of the annual report. It covers such areas as cover design, executive messages, scope of operation, financial narratives, charts, graphs, all types of art, photography, envelopes, paper stock, transmittal forms, methods of printing, and all other areas involved with the annual report.

"The Annual Report" also includes editorial matter and surveys as concerns budgets, responsibility for the preparation of such reports, distribution, production, and many other areas of significant interest. Other special sections include a glossary of terms used by those directly or indirectly involved with the annual report, special color charts, typography, terminology, and trademarks.

The volume is published by The Annual Report Division of Research Bureaus, Inc., Box 68, Kensington Station, Detroit, Michigan. The price of the volume is $15.00.

Instrument Society of America

1960 Conferences and Exhibits

The Houston, Texas Section of the Instrument Society of America will act as host to the first of ISA's three Instrument-Automation conferences and exhibits scheduled for 1960. The conference sessions will be held February 1–4 at the Rice Hotel, and exhibit will be staged in the Sam Houston Coliseum, February 2–4.

More than 60 papers will be presented at the conference and the exhibit, which occupy some 25,000 sq. ft. in the Coliseum, will be devoted to display and demonstration of the newest products of the nation's leading instrument manufacturers and their representatives.

Transducer Compilation

Working through the Instrument Society of America Primary Elements Committee, a special Transducer Committee will assemble, classify, collate, edit, and publish a comprehensive compendium of up-to-date information on all known transducers used in industry, medicine, biology, military applications, etc. This program will embrace all transducers described in the literature and those commercially available, both foreign and domestic. A bibliography of source material and a listing of manufacturers of transducers will be kept continuously up-to-date and periodic publication of revised editions is planned. This information will eventually be entered into information retrieval systems to facilitate rapid determination on the availability of a transducer having specific characteristics.

A transducer is defined as any device which enables the conversion of any physical, chemical or biological phenomenon into an electrical, hydraulic, pneumatic, mechanical, optical, and other signal for transmission, recording, measurement, analysis, actuation, output, or control.

Compilation and publication of the first volume is expected in January 1961. Coordination of this project, including editorial and production activities, is under the direction of Charles W. Covey, editor, *ISA Journal.*

Sewage Plant Effluent

Betz Laboratories, Inc., consultants on industrial water problems, announce the publication of Technical Paper No. 138 entitled, "Problems in the Use of Sewage Plant Effluent for Boiler and Cooling Purposes."

This 16-page study reports on how sewage plant effluent can be used satisfactorily in recirculating cooling systems and as boiler make-up provided it is adequately treated to avoid operating problems such as organic growths, deposits and corrosion in cooling systems and foaming, carryover and ammonia corrosion in boiler and steam systems. The necessary treatment can become complicated and expensive due to interfering substances in sewage, and may well become uneconomical if an adequate alternate supply of fresh water is available.

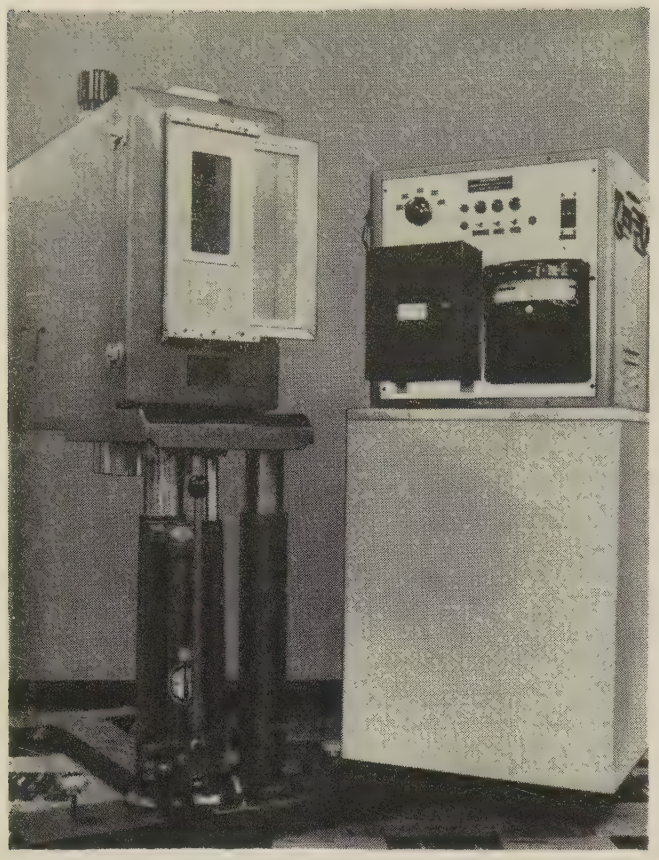
Copies of Betz Technical Paper No. 138 are available by writing to Betz Laboratories, Inc., Gillingham & Worth Streets, Philadelphia 24, Pa.

Fabric Research Laboratories

Extreme Temperature Chamber

A new apparatus facilitating the study of nonrigid materials at extreme temperatures has been announced by Fabric Research Laboratories, Inc., Dedham, Mass.

Called the FRL Extreme Temperature Test Chamber, the new apparatus is designed principally for use with Instron tensile testing instruments and makes possible tensile and



Extreme temperature test chamber (left) and control box (right)

compressional tests at a temperature range from −95°F. to +1000°F.

Self-contained on a rollaway carriage, the chamber can be used independently as an oven or cold chamber, with working space of about 3000 cu. in. Special chambers can also be constructed with large dimensions.

Interest in the behavior of textiles, plastics, rubber, wire, ceramics, etc., at these extreme temperatures includes not only their use in missiles or other devices which travel at ultra high speeds, but in numerous industrial applications as well.

Slush Pulp Consistency Determination

Rapid nondestructive measurement of per cent consistency of unbleached, softwood, slush pulp by the Model 104 nuclear magnetic resonance (NMR) analyzer is detailed in Material Study Report $28.58, published by Schlumberger Well Surveying Corp., Ridgefield Instrumentation Division.

Per cent consistency determinations made in 30 sec. by the NMR analyzer indicate a standard deviation of approximately ±0.05%. The report describes the steps in sample preparation and NMR testing, concluding with the presentation of calibration curves for slush pulp consistency.

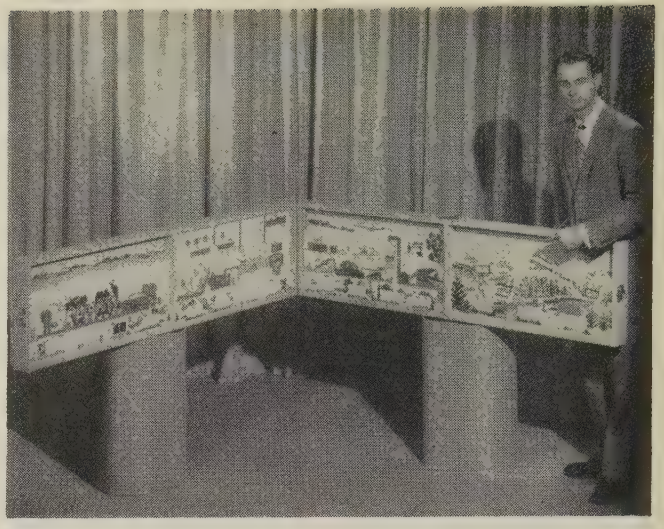
Copies are available from Schlumberger Well Surveying Corp., Ridgefield Instrumentation Division, Ridgefield, Conn.

Wisconsin Paper Industry

Papermaking Display

The Information Service of the Wisconsin Paper industry has developed a small, handy unit explaining the art of papermaking. The colored, electrically activated exhibit weighs about 45 lb. and extends to 12 ft. It is 15 in. high, 3¼ in. deep, and folds to 6 ft. in length.

With the help of the colored panels and various small turning machines, the display tells the story of paper from



Portable exhibit on the papermaking process, developed by the Information Service, Wisconsin Paper Industry

forests to finished product. It can be lent to schools, used as a speech prop, as a training aid, as an introduction to plan tours, etc.

Further information about the display can be addressed to the Information Service, Wisconsin Paper Industry, 104 N. Commercial St., Neenah, Wis.

Bibliography on Tall Oil Published

Covering subjects from abietic acid to zinc treatment, a new complete Tall Oil Bibliography discusses the preparation, properties, reactions, and applications of tall oil and its derivatives.

The Bibliography is published by The Institute of Paper Chemistry, under the sponsorship of the Tall Oil Products Division of the Pulp Chemicals Association.

Over 1600 articles, reviews, and patents are included in the Bibliography covering the literature to the end of 1957. Tall oil, an important by-product of the pinewood used in sulfate pulp production, has grown from a relatively unknown product to one of increasing value in commerce. Tall oil is a mixture of fatty acids and rosin acids. Tall oil fatty acids have a growing industrial usage in soaps and detergents, coatings and resins, and rubber chemicals as well as many other products and processes.

Production of crude tall oil in the United States is estimated to exceed 800 million pounds in 1959 against 310 million pounds in 1950. This represents an average annual growth, over the past decade, of approximately 11%.

The Bibliography is indexed by subjects, authors, and patents. Almost 1000 patents are indexed from the United States, Europe, and Russia.

Totaling 450 pages, this is the third edition of a bibliographical series initiated by the late Dr. J. West. It has been prepared under the direction of Jack Weiner of The Institute of Paper Chemistry. Copies have been supplied to members of The Institute of Paper Chemistry and to the Pulp Chemicals Association. Nonmembers may purchase copies from The Institute or request copies, on company letterheads, from the Pulp Chemicals Association, 60 East 42nd St., New York 17, N. Y.

The Ufan Continuous Digester Pilot Plant*

The UFAN digester system is designed to produce kraft, semichemical, or acid sulfite pulps. It is expected that fully cooked kraft pulps will require 30 to 35 min. total cooking time, high yield kraft will be cooked in 15 to 25 min., and

* Pulp and Paper Laboratory, Chemical Engineering Department, University of Florida, Gainesville, Fla.

semichemical pulps in 30 to 45 min. Acid sulfite pulps should require about 60 min.

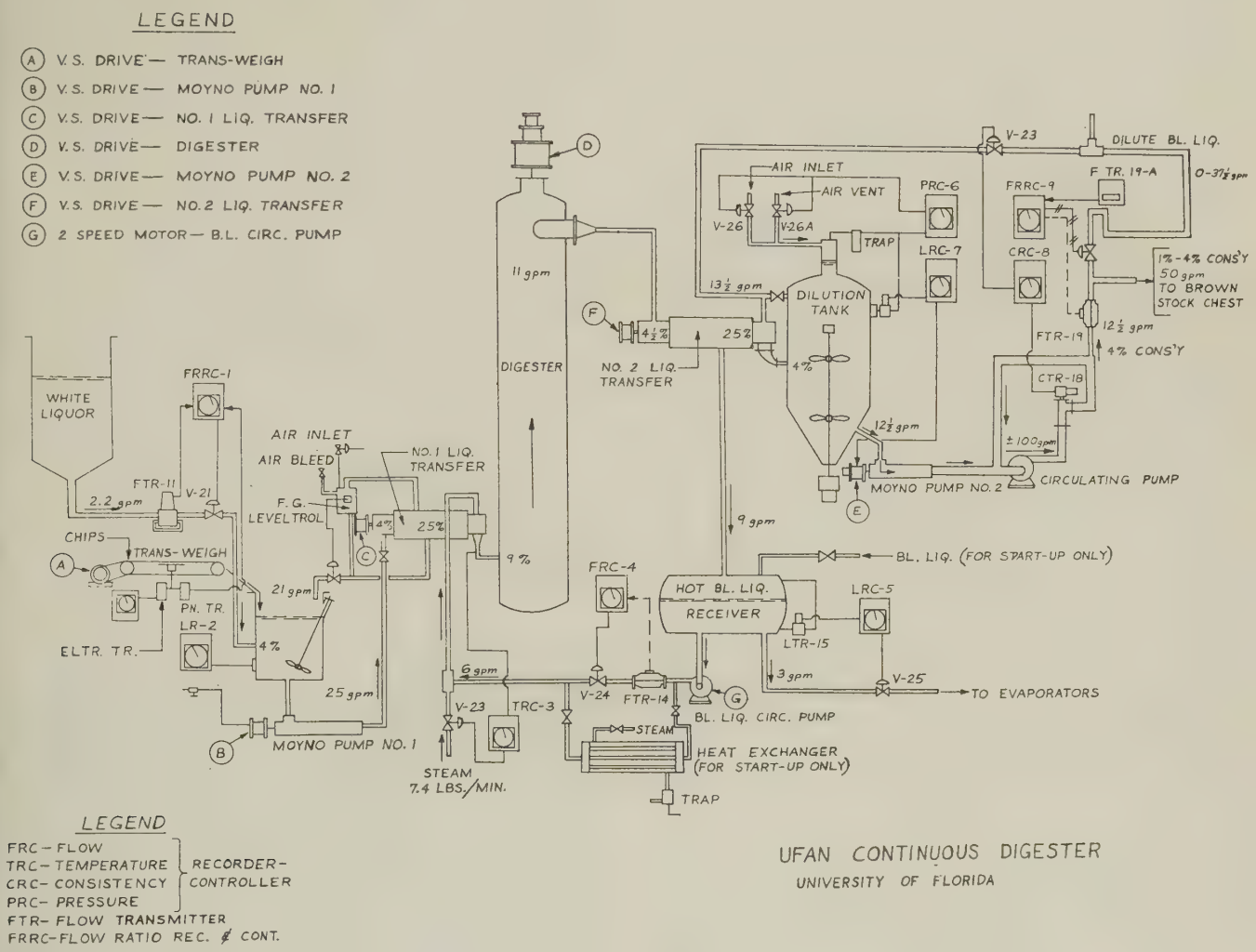
The process itself is covered by U. S. patent application no. 578,378 and the operating principles of the digester are covered by U. S. patent application no. 770,424.

The principles of operation involve, first, the transportation of shredded chips and a large excess of white liquor through a pump to develop a hydrostatic pressure greater than the steam pressure to be used in the digester. Second, most of the white liquor, the chip conveying medium, is expressed from the chips just before entering the digester and is replaced by recirculated black liquor, already at full digester temperature. Sufficient of this recirculated liquor is provided to insure a very high liquor/wood ratio.

and is replaced by cooler liquor pumped from the recovery system. Since this new mixture will be at 190°F. or lower, steam pressure is lost. Therefore, this steam pressure is replaced by a balancing air pressure to insure steady, slow flow throughout the system. The final pulp suspension, under this air pressure, is pumped out of the system, the pump serving as a nonclogging, pressure-reducing valve.

The pilot plant installation is designed to produce 3.0 ovendry tons of fully cooked pulp per day. The accompanying flow sheet can be used to explain the operation.

Shredded chips are fed to the system on a belt conveyor equipped with a Transweigh Beltmeter. The electrical impulse from the load cell of the weighing device is transmitted to a Minneapolis-Honeywell recorder. In addition to



The UFAN continuous digester pilot plant

This high liquor/wood ratio is very important. It provides a highly fluid condition in the digester in contrast to the usual chip packing. This fluidity insures absolutely uniform temperature throughout and identical cooking conditions for all chips passing through the digester. It also permits the use of a low concentration of alkali even at the start of the cook, required by the fact that the incoming chips encounter maximum temperature.

The high liquor/wood ratio and the low upward velocity of liquor through the digester would allow chips to settle if unchecked. Therefore, the digester is provided with a slowly rotating screw which moves chips upward at the same velocity as the screw.

After cooking, excess liquor is expressed from the chips

recording, this instrument is equipped with a pressure transmitter. This pressure transmitter control the flow of white liquor from white liquor storage so that 10 lb. of white liquor are supplied per pound of chips. This control is accomplished through Fischer-Porter instruments FRRCl and FTR-11 and Fischer Governor valve V21.

Chips and liquor are kept in uniform suspension at 4% consistency by a Lightnin Mixer. A Moyno pump takes the suspension from the bottom of the tank and develops 150 p.s.i. hydraulic pressure on the system, moving the suspension into the no. 1 liquor transfer screw. The level in the tank is recorded by Fischer-Porter level recorder, LR-2.

This transfer screw, a specially designed screw press, increases the consistency from 4 to 25%. The pressure

casing around the barrel of the screw insures that full hydraulic pressure will be maintained on the expressed liquor. A Fisher Governor Leveltroller, with balancing air pressure above the liquid level in the float chamber, automatically bleeds off the excess liquor without loss in hydraulic pressure.

Recirculating black liquor is pumped to the discharge of the transfer screw by a Duriron Co. high head pump. The high head feature of the pump is used only at start-up, in conjunction with the heat exchanger shown in the figure. (This heat exchanger is not used in the pulping operation.) During pulping operations the pump is operated at half speed, supplying low head. The black liquor line is equipped with a Fischer-Porter magnetic flow meter, FTR14 and recorder-controller FRC-4 which controls the flow, through Fisher Governor valve V24, to a preset value.

Sufficient steam is supplied to heat the chips and the 3 lb. of white liquor per pound of chips to cooking temperature. No steam is required for the recirculating black liquor, already at full temperature. A thermometer bulb at the entrance to the digester actuates Fischer-Porter temperature recorder controller TRC-3 and Fischer Governor valve V-23.

Chips, at 9% consistency, are transported up through the digester by a screw rotating at $^1/_6$ to $^1/_2$ r.p.m., depending on cooking time. Helix angle and speed of rotation are designed so that chips will be carried upward at the same speed as the cooking liquor.

Cooked pulp is discharged at the top of the digester as shown. Not shown in the flow-sheet, is a bleed-off at the top of the digester for removal of turpentine and other volatiles. The pulp, after leaving the digester, loses its excess liquor in the no. 2 liquor transfer screw, identical in design to the no. 1 screw already described. The excess liquor flows, under full steam pressure, to the hot black liquor receiver. Bleed-off of the excess over that required for recirculation is accomplished automatically by Fischer-Porter level recorder-controller LCR-5 and LTR-15, operating Fisher Governor valve V-25. This hot liquor would normally be pumped to the evaporators at full steam pressure without the usual flashing to atmospheric pressure and a low temperature.

After being pressed to 25% consistency in the no. 2 transfer screw, the pulp is diluted to 4% consistency with dilute black liquor from the pulp washers. The no. 2 transfer screw may be considered as the first stage of washing. For every pound of pulp, 24 lb. of dilute black liquor at about 170°F. mixes with 3 lb. of cooking liquor at about 365°F., resulting in a mixture at about 190°F. This mixture is kept in smooth suspension in the dilution tank by a Lightnin Mixer entering at the bottom.

Air pressure is applied at the dilution tank to counterbalance the steam pressure in the digester. Level is maintained by Fischer-Porter level recorder-controller LRC-7. Control of this level in the pilot plant, will be manual temporarily; an operator manipulating a vernier rheostat on the d.c. motor drive of Moyno pump no. 2. Air pressure is controlled by the Fischer-Porter pressure recorder-controller PRC-6 and Fisher Governor valves V-26 and V-26A.

The pulp flows through the Moyno pump, where its pressure is throttled, through a circulating pump to Fischer-Porter consistency controller-recorder, CRC-8 and CTR-18. (This circulating pump is used in the pilot plant only because normal pulp volume is too low for accurate consistency control. The pump by-pass provides the necessary volume for good control.) The consistency controller automatically provides the correct volume of dilute black liquor, through Fischer Governor valve V-23, to insure that consistency in the dilution tank will be 4.0%.

Volume of pulp flow is measured and recorded with Fischer-Porter magnetic flow meter and flow ratio recorder-controller, FTR-19, FRRC-9, and FTR 19A. Exit consistency of the pulp can be set at any value below 4%.

It is apparent the synchronization of speeds of the various units is essential. Belt speed of the chip conveyor, speeds of Moyno pump no. 1, transfer screw no. 1, digester screw, transfer screw no. 2, and Moyno pump no. 2, must all be operated in fixed ratios, one to the other. If one unit is changed in speed all others must change in proportion. Therefore, all six motors on these drives are Westinghouse d.c. motors. The main rheostats of all six motors are mounted on a single shaft to provide this synchronization. In addition, a vernier rheostat and tachometer are provided for each motor in order that small adjustments in speed can be made. Source of power for the d.c. motors is a Westinghouse AVR power unit.

It will be apparent that the instrumentation makes it possible to compute pulp yield accurately at any time during the operations. Weight of chips supplied can be read from the recorder on the Transweight conveyor. Samples of chips will be taken continuously and periodic moisture determinations will be made. The consistency controller and magnetic flow meter will give continuous data on weight of oven-dry pulp produced. Therefore, calculation of yield becomes a simple matter.

This pilot plant is now being installed at the University of Florida Pulp and Paper Laboratory. As soon as operations have been thoroughly checked in several trial runs the unit will be displayed as part of the next Pulping Conference at the University. Goslin-Birmingham Manufacturing Co., of Birmingham, Ala. is the manufacturer of the digester, transfer screws, etc., and will manufacture and sell the commercial units developed from the pilot plant operation.

OBITUARIES

Thomas W. Bauer

Thomas W. Bauer, physicist, at the Central Research Division of Abitibi Power and Paper Co., Ltd., passed away suddenly at Sault Ste. Marie, Ont., on Nov. 7, 1959.

Dr. Bauer was born in Innsbruck, Austria, in 1921 and graduated from McGill University in 1944 and Ohio State University in 1950.

He joined Abitibi's Central Research Division in April. 1951, and during the past years has made several important contributions to the industry in the fields of physics and statistical analysis. He became a member of TAPPI in 1955.

He is survived by his mother, Mrs. Edith Bauer, and a sister, Mrs. Eva Dimand.

Clifford Roy Swett

Clifford R. Swett, vice-president and technical director of the National Wax Co., Skokie, Ill., passed away Dec. 5, 1959.

Mr. Swett was born Aug. 19, 1904, in Dixfield, Me. He graduated from Pratt Institute in 1927 as an Industrial Chemical Engineer and worked from 1921 to 1938 for the Oxford Paper Co., Rumford, Me., Albermarle Paper Manufacturing Co., Richmond, Va., Escanaba Paper Co., Escanaba, Mich., and the Container Corporation of America in Philadelphia and Chicago. In 1938 he joined the Technical Service Department of Stein Hall Mfg. Co. which he left in 1945 to become technical director of National Wax Co. with which company he has been associated for the last 15 years.

Mr. Swett became a member of the Technical Association of the Pulp and Paper Industry in 1929.

TAPPI DIVISIONS AND COMMITTEES

Reports of Activities

Optical Properties Committee

A meeting of the Optical Properties Committee was held in Portland, Ore., on Aug. 18, 1959. Present were:

R. S. Hunter, *chairman*, Hunter Associates Laboratory, McLean, Va.
H. C. Brill, *asst. secy.*, Du Pont, Chestnut Run, Wilmington, Del.
R. M. Mehta, Tribini Tissues Ltd., Calcutta, India
D. F. Jenkins, Champion Paper & Fibre, Hamilton, Ohio
Yas Nomiyama, St. Regis, Carthage, N. Y.
W. A. Wink, Institute of Paper Chemistry, Appleton, Wis.
J. A. Van den Akker, Institute of Paper Chemistry, Appleton, Wis.
M. P. Boland, Columbia-Southern Chemical Corp., Barberton, Ohio
W. H. Cuffey, Kimberly Clark Corp., Neenah, Wis.
Martin Nill, Longview Fibre Co., Longview, Wash.
P. J. Pernice, General Dyestuff Co., Portland, Ore.
S. M. Chapman, Pulp & Research Institute of Canada, Montreal, Que.
J. E. Jeffery, Rayonier, Inc., Shelton, Wash.
Gene Baldwin, Champion Paper & Fibre, Hamilton, Ohio
Kay Bredleau, Crown Zellerbach Corp., Camas, Wash.
Bob Wood, Crown Zellerbach Corp., Camas, Wash.
Stewart Rowe, P & G, Cincinnati, Ohio
Ed. Brandon, Miami University, Oxford, Ohio
H. G. Jansen, Simpson-Lee Paper Co., Everett, Wash.

Mr. Hunter noted that Optical Properties Committee has no members from the West Coast. He invited all guests to join.

Optical Properties Committee meeting held at Portland, Ore., August 18, 1959. (Left to right). 1st row: R. H. Menta, Tribini Tissues Ltd., Calcutta, India; R. S. Hunter, Hunter Associates Lab., McLean, Va.; H. C. Brill, E. I. du Pont, Chestnut Run, Wilmington, Del. 2nd row: D. F. Jenkins, Champion Paper & Fibre Co., Hamilton, Ohio; Yas Nomiyana, St. Regis Paper Co., Carthage, N. Y.; W. A. Wink, Institute of Paper Chemistry, Appleton, Wis.; J. A. Van Den Akker, Institute of Paper Chemistry, Appleton, Wis. 3rd row: J. E. Jeffery, Rayonier, Inc., Shelton, Wash.; S. M. Chapman, Pulp & Paper Research Institute of Canada, Montreal, Que.; P. J. Pernice, General Dyestuff Co., Portland, Ore.; Mat Boland, Columbia-Southern Chemical Corp., Barberton, Ohio; W. H. Cuffey, Kimberly Clark Corp., Neenah, Wis. 4th row: G. Baldwin, Champion Paper & Fibre Co., Hamilton, Ohio; B. Wood, Crown-Zellerbach Corp., Camas, Wash.; M. Nill, Longview Fibre Co., Longview, Wash.

The minutes from the last meeting were read and approved.

Mr. Hunter issued an invitation to members to report new optical tests to the Routine Control Methods Committee.

Committee Projects

1. Project 144—A. G. Lang—Illuminants for Color Matching. In the absence of Mr. Lang, Mr. Hunter reported that work is just starting on a TAPPI Standard for artificial illumination.

2. Project 321—W. A. Wink—Gloss Measurement. Shooting for ±1% tolerance between instruments. Method is nearly ready for submission. Project will be completed when method is submitted and accepted.

3. 564 Brightness of Paper and Paperboard—Van den Akker—Reported in *Tappi*. Now ready for submission. This will be 1959 revision of present method. This part of project will be completed when method is submitted.

Task force reestablished with Dr. Van den Akker as chairman of the new project to bring the standard into conformity with recent changes in spectral response developed at the Institute of Paper Chemistry. Moved, seconded, and passed.

4. 565 Opacity of Paper—Van den Akker—Published in *Tappi*, but not in final form. Project will be completed when published in final form.

5. 566 Preparation of Method for Use of Chapman Smoothness Tester. It was suggested that Dr. Zettlemeyer and Miss Fetsko of Lehigh University be invited to sit in on optical meetings.

Printing quality, smoothness, and formation were of great interest in replies to questionnaire. Some time was taken in a discussion on the sphere of influence of the optical committee. Mr. Hunter announced that a joint meeting would be held on Thursday at 11:00 a.m. to pick up possible tests from other committees.

Proposed Committee Projects

1. *Formation Testing.* Mr. Obenshain stated that this project is now inactive. Dr. Van den Akker outlined the difficulty of measuring formation. Believed to be too early to standardize. The discussion indicated lack of goal.

2. *Transparency—Show Through.* The discussion centered around the difficulties of setting up tests. A possible overlap with the Graphic Arts Committee was also pointed out. It was suggested that we cooperate, with Graphic Arts leading the way. It was suggested that Mr. Hunter write to the chairman of Graphic Arts and offer to cooperate.

3. *Optical Brighteners.* There were two papers on this subject in the Portland program. General Analine has developed instrument for measuring fluorescence on both the wire and felt side of the sheet. Eastman Kodak is using a modified Beckman DK-2 Spectrophotometer. Hunter flips the ultraviolet-absorbing filter from incident to sample-viewing light beam in his tristimulus reflectometer.

There is need for a new standard for measuring the effect of optical brightness in paper. Motion (Dr. Van den Akker) a task force be set up for determining the need for a method for measuring the contribution of the optical dye to the brightener. Seconded and passed.

Following instructions of the Steering Committee, we went over our methods, ten years old or older, to determine whether action on them is needed.

T 216 m-47, Color of Pulp—No change recommended.
T 217 m-48, Brightness of Pulp—To be revised by new task force.
T 442 m-47, Color of Paper—A footnote is needed to point out inapplicability to papers containing fluorescent dyes.
T 633 m-50, Magnesium Oxide Standard of Reflectance—Ten years not up. Dr. Van den Akker stated that present method is undesirable, but should not be replaced until a better one is secured.

August 20 Meeting

The following people were present at a brief meeting on Aug. 20, 1959, to hear reports of requests for cooperation from product committees.

R. S. Hunter, *chairman*, Hunter Associates Laboratory, McLean, Va.
H. C. Brill, *asst. secy.*, Du Pont, Chestnut Run, Wilmington, Del.
Stewart Rowe, Procter & Gamble, Cincinnati 39, Ohio
D. F. Jenkins, Champion Paper & Fibre, Hamilton, Ohio
Yas Nomiyama, St. Regis, Carthage, N. Y.
W. A. Wink, Institute of Paper Chemistry, Appleton, Wis.

Steering Committee met and Hunter reports:

1. *Request from Paperboard Committee*—That we work on photographic method of measuring contour and surface smoothness by incidence of light at grazing angle. The chairman will ask John G. Cella of Lowe Paper to serve as our representative on task force to be set up.

2. *Pigments Committee*—Arranging new method for measuring the brightness of paper fillers, inclusive method. Optical Committee would like to monitor this before publication.

3. *Vanceometer*—Sears, The Institute of Paper Chemistry, and Fetsko have worked on correlation of instrument with printing. Hunter to invite Sears to work on task force. Question for optical group: What is proper light source and arrangment?

Mr. Hunter called attention of all TAPPI people to five ASTM published methods for color-difference measurement.

	Color Difference Determined by Using
D 1260–55T	Hunter Multipurpose Reflectometer
D 1365–55T	Hunter Color Difference Meter
D 1482–57T	GE Spectrophotometer
D 1495–57T	Color Eye
D 1536–58T	Colormaster Differential Colorimeter

Task Force Personnel

Current:
144—Illuminants (Lang, *chairman*, Vincent, Paris)
321—Gloss (Wink, *chairman*, Vincent, Obenshain)
564—Brightness (Van den Akker, *chairman*, Wing, Elsevier)
565—Opacity (Van den Akken, *chairman*, Silard, Hunter)
566—Chapman (Obenshain, *chairman*, Chapman)

New Projects:
Optical Brighteners (Patek, *chairman*, Van den Akker)
Paperboard Examination by Grazing Light (Cella, Hunter)
Vanceometer (Sears, Hunter)

H. C. Brill, *Assistant Secretary*

Mechanical Pulping Committee

A meeting of the Mechanical Pulping Committee was held at the Norton Co. Laboratory Conference Room, Worcester, Mass., on Oct. 5, 1959, with the following in attendance:

J. K. Kirkpatrick, *chairman*, Bowaters Southern Paper Corp.
S. R. Parsons, *first vice-chairman*, Consolidated Water Power & Paper Co.
Maung Shwe Htoo, *second vice-chairman*, International Paper Co.
J. H. Perry, *secretary*, Norton Co.
K. L. Pingrey, Diamond Gardner Corp.
F. W. O'Neil, New York State College of Forestry
Christian Anker, Risor Trasmassefabriker, Norway
J. Blinke, Procter and Gamble Co.
A. A. Yankowski, Kimberly-Clark Corp.
E. H. Johnson, Stevens and Thompson Co.
A. J. Winchester, TAPPI Headquarters

The meeting was opened promptly at 9:30 a.m. by J. K. Kirkpatrick, chairman. The roll call was taken and those present are noted above. The roll call pointed up the fact that certain members were absent and that these same people were not present at the last meeting. This prompted a motion by Earl Johnson which was seconded by Fred O'Neil and amended by Tony Yankowski that the first vice-chairman, S. R. Parsons, should write these delinquent members, reminding them of their obligation to attend meetings, and if they or their delegates are not able to do so, without just cause, that they be asked to resign from the committee. It was emphasized during the discussion regarding this motion that the majority of the members of the committee were most active and conscientious in fulfilling the duties assigned to them; in other words, the Mechanical Pulping Committee is a working committee and all members share equally in the work assignments.

The chairman read a letter from M. S. Anderson of the Oxford Paper Co. in which he tendered his resignation be-

Combined meetings of the TAPPI and CPPA Mechanical Pulping Committees, held at Worcester, Mass., Oct. 5–6, 1959. Front row (left to right): W. H. de Montmorency, Pulp & Paper Research Institute of Canada, Montreal, Que.; K. L. Pingrey, Diamond Gardner Corp., Ogdensburg, N. Y.; A. J. Winchester, TAPPI, New York, N. Y.; G. M. Alarie, Richmond Paper Co., Ltd., Bromptonville, Que.; J. H. Perry, Norton Co., Worcester, Mass.; C. R. Powell, E. B. Eddy Co., Ltd., Hull, Que.; M. S. Htoo, International Paper Co., Glens Falls, N. Y.; M. A. Christie, Norton Co., Worcester, Mass. Back row (left to right): I. M. McArthur, Canadian International Paper Co., Trois-Rivieres, Que.; F. W. O'Neil, State University of New York, Syracuse, N.Y.; S. R. Parsons, Consolidated Water Power & Paper Co., Wisconsin Rapids, Wis.; R. M. Murley, Bowaters Paper Mills, Cornerbrook, Nfld.; C. Anker, Risor Traemassefabriker, Risor, Norway; J. K. Kirkpatrick, Bowaters Southern Paper Corp., Calhoun, Tenn.; T. M. Cole, Norton Co., Worcester, Mass.; H. P. Richards, Ontario Paper Co., Thorold, Ont.; J. W. Holloway, Price Bros. & Co., Kenogami, Que. Middle Row (left to right): J. Blinka, Procter & Gamble, Cincinnati, Ohio; E. C. Mason, Abitibi Paper Co., Ltd., Iroquois Falls, Ont.; T. W. Dobson, Donnacona Paper Co., Donnacona, Que.; W. Y. Benn, Consolidated Paper Corp., Grand' Mere, Que.; A. A. Yankowski, Kimberly-Clark Corp., Neenah, Wis.; E. H. Joynson, Stevens & Thompson Paper Co., Greenwich, N. Y.; A. L. Flora, Anglo-Canadian Paper Mills, Quebec, Que.; S. E. Steeves, Anglo-Newfoundland Development Co., Ltd., Marathon, Ont.; W. H. Copeland, Norton Co. of Canada, Ltd., Hamilton, Ont.

cause of the pressure of new responsibilities in his company. The committee refused to accept the resignation because M. S. Anderson's past record shows that he is most valuable to the committee and that his assignments have been completely fulfilled. It was decided that M. S. Anderson would remain on the committee, with the hope that if he is not able to attend, he will arrange to have his alternate do so, as provided in our bylaws.

The names of T. F. LaHaise, Ryegate Paper Co., and Joseph Blinka, Procter and Gamble Co., were proposed as members. Both were unanimously accepted.

The minutes of the last committee meeting held in New York on May 28, 1959, were read by the secretary and were accepted as read.

Project 678—The Effect of Closed White Water System on Groundwood Brightness—F. W. O'Neil, Chairman

F. W. O'Neil reported that this project, which had been TAPPI Grant No. 162, was concluded. The cost of carrying out this grant amounted to more than $600 which had been allotted; however, the additional cost was absorbed by the State University of New York, College of Forestry. A copy of Mr. O'Neil's report to TAPPI was circulated. He pointed out that this had been prepared by F. W. Lorey, Pilot Plant Group Leader, who is also Assistant Professor of Pulp and Paper Technology at the University, and E. C. Jahn, Associate Dean for Research. The report will be published in *Tappi*. There was much discussion on this subject and the question was raised: should this project be considered final? It was concluded that the report covered all the pertinent facts and that further study would be considered in the future.

Project 636—Effect of Underwater Storage of Pulpwood on Groundwood Brightness—Karl E. Pingrey, Chairman

The third interim report was submitted by K. E. Pingrey and copies were distributed to all members. This report will be published in *Tappi*. Mr. Pingrey pointed out that there did not seem to be a definite trend being established from these data. In his opinion the most significant thing was that peeled, stored wood did not seem to lose brightness, and it generally regained brightness better than unbarked wood which had been stored. He mentioned also that all determinations had been carried out on the same pulpstone, one which had run only part of the time, and that only fresh water was used on the showers and that the temperature was always the same. Because of all this, he was unable to explain the variations shown in the data. There seemed to be cycles where there were gains in brightness from time to time. He did not feel that results were conclusive so intends to carry out a final test before the river freezes in the winter. Mr. Anker reported that he had made some determinations along these lines, also getting variations up and down in brightness and he, too, reported that there was a gain in the manganese content as did Mr. Pingrey. In both cases, the manganese content was very much above what one might expect and it was concluded that this must come from the wood and is cumulative. Mr. Anker even went to the extent of draining the water shed from which he obtains fresh water and cleaning out the ponds or reservoirs. Further determinations will be carried out in the Risor mill because a new system has been instituted in which there is absolute control over the amount of fresh water in the system. A further report on this subject will be forthcoming from Mr. Anker.

Project 591—Groundwood Manual—E. H. Johnson, Chairman

The manual has been edited by Mr. Yankowski and Mr. Parsons and is now in the hands of Mr. Winchester for publication.

Project 779—A Manual Giving Descriptions of the Various Processes of Mechanical Pulping—J. H. White, Chairman

A motion was made by Earl Johnson and seconded by Maung Htoo that the objective of this project be clarified to read as follows: "To publish annually in *Tappi* magazine (December issue) a résumé of new developments in the mechanical pulping field, including all matters within the scope of the Mechanical Pulping Committee." This motion was carried. John White wrote a letter to Mr. Kirkpatrick in which he asked to be relieved of his duties as chairman of this subcommittee. The reasons for his request were that his mill is undergoing extensive alterations, both in equipment and process, which will require his full attention for many months. He also mentioned that he has additional responsibilities in respect to wood procurement. In view of this, he pointed out that he could not do justice to the subcommittee and, in fairness, asked to be relieved. However, he will continue to be a member of the committee and will do all that time permits to help on this project and all others. Mr. Kirkpatrick expressed grateful appreciation for all that John has done and regretfully accepted his resignation from the subcommittee. He then appointed Joe Blinka chairman, and Tony Yankowski volunteered to render all assistance possible to this subcommittee. Christian Anker was appointed subchairman to work with Mr. Bersano on the European phase of this project.

Project 680—A Survey of Groundwood Mill Process Instrumentation—J. K. Kirkpatrick, Chairman

Mr. Kirkpatrick had nothing to report at this time on this project, but will submit a report at the next meeting. There was considerable discussion on this subject and it was pointed out that very little had been done to acquaint the instrumentation industry with the opportunities for instrumentation in groundwood mills. It was felt that much more could be done of value to the industry if problems were presented to the instrument people for solution. J. H. Perry was requested to write to the chairman outlining possible suggestions for further groundwood instrumentation, this letter to be augmented by suggestions from other members.

Maung Shwe Htoo, program chairman for the winter meeting, reported that he had circulated a questionnaire in July in which he had solicited material for papers to be presented in February. This has resulted in four good prospects. In order that a complete program be assured, Maung Htoo is investigating several other excellent and interesting possibilities. It was concluded that a suitable program would be presented in February.

It was decided that the next meeting of this committee will be held in New York at the Commodore Hotel sometime during the annual meeting. Mr. Kirkpatrick will inform the members of the exact time and location.

The Fourth International Mechanical Pulping Conference Committee, which had been appointed at a joint meeting, was ratified unanimously by the Committee. Christian Anker proposed that the Fifth International Mechanical Pulping Conference be held in Europe. Among the many advantages, he pointed out, would be increased participation by Europeans and that it would serve to cement relations generally throughout the groundwood industry, and also that Europeans would be induced to present papers on developments in their countries which would not otherwise be available to our industry.

All members agreed that Mr. Anker's suggestions had much merit and the committee will act upon Mr. Anker's proposal when plane are being formulated for the Fifth International Conference.

Mr. Winchester mentioned that requests for research

appropriations should be ready for presentation before April 1, and he emphasized that members should think of projects requiring financial assistance, and should be ready to present them at the next meeting in February. The chairman requested that the secretary remind members by letter within a month. A recess was declared at 3:50 p.m. and the meeting resumed at 4:00 p.m.

Christian Anker acquainted the committee with the fact that he has set up his mill system so that he has, for research purposes, one grinder complete with screens and other equipment which enables him to make a complete evaluation. He intends to conduct some research on aspen production and later will grind birch groundwood. He will report on his research efforts to the committee.

A motion was made by Maung Htoo and carried that the committee review its activities for the past 10 years, in order that there may not be a duplication of effort and also that some of the past projects might be worthy of further investigation. A. J. Winchester and J. H. Perry agreed to make this review at the first opportunity.

A number of suggestions were offered for further study by the committee, among them, S. R. Parsons suggested that a suitable subject would be wood moisture effect on pulp quality.

E. H. Johnson suggested several interesting subjects: a device to measure wood moisture such as the beta gage, the effect of fines on brightness, mechanics involved in bleaching, and cost versus loss of fines. A. A. Yankowski proposed that the mechanism of breakdown of hydrosulphite in bleaching could be clarified. A. J. Winchester suggested that an improved micrometer lathe or better method of burring be devised. He also suggested that an accurate means of measuring burr penetration would be of great value to the industry.

The meeting adjourned at 4:30 p.m.

J. H. Perry, *Secretary*

Plastics Committee

The 1959 fall meeting of the TAPPI Plastics Committee was held at the Edgewater Beach Hotel in Chicago on Sunday, September 20th at 2:00 p.m. This meeting preceded the Fourteenth Annual Plastics Paper Conference held at the Edgewater Beach Hotel.

The following members and guests were present:

W. N. Stickel, *chairman*, Texon, Inc., South Hadley Falls, Mass.
H. A. Arbit, Union Carbide Plastics, Bound Brook, N. J.
R. E. Benton, Armour Alliance Industries, Alliance, Ohio
R. L. Erratt, The Dow Chemical Co., Midland, Mich.
J. J. Forsythe, Jr., International Paper Co., Niagara Falls, N. Y.
T. A. Howells, Institute of Paper Chemistry, Appleton, Wis.
H. G. Maeder, Jr., Dixie Cup Co., Easton, Pa.
G. Pixler,* Colton Chemical Co., Cleveland, Ohio
J. C. Pullman, American Cyanamid Co., New York 20, N. Y.
F. Rosenthal, Knowlton Brothers, Inc., Watertown, N. Y.
R. L. Savage, Naugatuck Chemical Division, Naugatuck, Conn.

Chairman Wes Stickel introduced Mr. Loheed and Mr. Benton, respectively, as the new vice-chairman and secretary of the Plastics Committee.

Chairman Stickel then read a letter from W. A. Schenck who submitted his resignation as chairman of Tack Force Project 586 dealing with "Adhesion of Polyethylene Coating to Paper" due to pressure of new responsibilities. Chairman Stickel read Dr. Schenck's letter outlining suggestions for presenting a more effective proposal to the TAPPI Appropriations Committee. Mr. Stickel then read the Appropriations Committee's reasons for rejecting the previous proposal for Project 586.

* Guests.

Plastics Committee meeting, held at the Edgewater Beach Hotel, Chicago, Ill., Sept. 20, 1959. Standing (left to right): G. Pixler, Colton Chemical Co., Cleveland, Ohio; R. L. Erratt, Dow Chemical Co., Midland, Mich.; H. A. Arbit, Union Carbide Plastics, Bound Brook, N. J.; J. J. Forsythe, International Paper Co., Niagara Falls, N. Y.; F. Rosenthal, Knowlton Brothers, Inc., Watertown, N. Y.; R. L. Savage, Naugatuck Chemical Division, Naugatuck, Conn. Seated (left to right): R. E. Benton, Armour Alliance Industries, Alliance, Ohio; W. N. Stickel, Texon, Inc., South Hadley Falls, Mass.; H. G. Maeder, Jr., Dixie Cup Co., Easton, Pa.; T. A. Howells, Institute of Paper Chemistry, Appleton, Wis.

Since no new chairman could be immediately selected for Project 586, no definite decisions could be made regarding the future of this project. It was suggested by T. A. Howells that a round robin system among committee members on testing procedures be developed. This would demonstrate the interests of the committee in this project before any future request for TAPPI financing.

V. T. Stannett summarized the progress made on the Permeability Project. Dr. Stannett revealed that effective methods have been developed to measure water pick-up of polyethylene and other hydrophobic films throughout the entire humidity range. The results of this method correlates closely with similar data obtained in a project by Bell Telephone Laboratories. The reports made at the 14th Annual Plastics-Paper Conference finished this project.

The motion was made by Mr. Maeder and seconded by Mr. Arbit that the committee recommend that Research Project 120 on Permeability be published in the form of a TAPPI Monograph. This motion was unanimously passed.

Dr. Stannett informed the committee that approximately $6000 worth of equipment is available from the permeability project. This equipment can be used on any future project dealing with film absorption or permeability. Mr. Maeder recommended that Dr. Stannett keep the Plastics Committee advised regarding areas of study where this equipment would be of value. This would aid the committee in decisions on future projects. One possible area would be the effect of oxidation on polyethylene on absorption and permeability of water.

Under New Business, Chairman Stickel recommended that a meeting be held in New York among the groups involved selecting test methods for plastics-paper combinations. R. W. Loheed has submitted written recommendations regarding groups which should be invited to participate in the proposed conference. The purpose of this meeting would be to clarify the responsibilities of the various groups in this field. It was the consensus among the committee members present that we should limit thes cope of our committee activities to limited areas of testing, e.g., barrier tests, structural tests, and possibly organoleptic tests.

F. Rosenthal and W. Stickel agreed to form the nucleus of a group to work on methods of testing internal bond strengths. Messrs. Maeder, Stannett, and Howells volunteered to aid in this study.

R. L. Savage, Naugatuck Chemical Co., agreed to be chairman for a group studying test methods for resin identification and content in paper. G. Pixler of Colton Chemical Co. said that R. N. Campen of Colton Chemical Co. would assist in this study.

During the general discussion, questions were raised on means to coordinate activities of committees to prevent duplication of interest. It was suggested by Mr. Pixler that TAPPI National Headquarters be requested to publish a summary of committee projects to serve as guides for the various committee meetings. It was also suggested by Mr. Pixler that TAPPI Headquarters also furnish the committees with available information on interests and activities of groups outside of the TAPPI organization. These could be periodically supplied to the various committee chairmen as a summary of joint interests and overlapping interests among various TAPPI committees and outside groups.

It was suggested by Mr. Arbit that because of the wide scope of interests among the Plastics Committee members, that various subcommittees based on mutual interests be formed. These could be subcommittees on beater addition, polyethylene extrusion, saturation, etc. These subcommittees could then meet independently of the parent Plastics Committee.

R. E. Benton, *Secretary*

Chemical Methods Committee meeting held at Portland, Ore., Aug. 18, 1959. Left to right: L. C. Aldrich, West Virginia Pulp & Paper Co., Covington, Va.; J. C. Cousins, Penobscot Chemical Fibre Co., Great Works, Me.; R. B. Chamberlin, Fibreboard Paper Products, Inc., San Francisco, Calif.; K. H. Williams, Mid-States Gummed Paper Co., 6850 S. Harlem Ave., Argo, Ill.; A. E. Peterson, Crown Zellerbach Corp., Camas, Wash.; H. J. Sonntag, Crown Zellerbach Corp., Camas, Wash.; A. Stonis, Celulosa de Chihuahua, S.A., Anahuac, Chih, Mexico; J. T. Henderson, Crown Zellerbach Corp., Camas, Wash.; P. H. Flaherty, The Crossett Co., Crossett, Ark.; J. d'A. Clark, Corvallis, Ore.; S. J. Hazard, Jr., State University College of Forestry, Syracuse, N. Y.; V. H. Truesdale, Consolidated Water Power & Paper Co., Appleton, Wis.

Chemical Method Committee

The Chemical Methods Committee met at 1:30 p.m., Tuesday, August 18, at the TAPPI 10th Testing Conference, Multnomah Hotel, Portland, Ore.

The following were present:

John C. Cousins, Penobscot Chemical Fibre Co.
Aldon E. Peterson, Crown Zellerbach Corp.
Hans J. Sonntag, Crown Zellerbach Corp.
Antanas Stonis, Celulosa de Chihuahua, S. A.
Patrick H. Flaherty, The Crossett Co.
John T. Henderson, Crown Zellerbach Corp.
James d'A. Clark, *chairman* of Standards Committee
Sherrill J. Hazard, Jr., Consolidated Water Power
Vernon H. Truesdale, Consolidated Water Power
R. Bruce Chamberlin, Fiberboard Paper Products, Inc.
C. E. Brandon, *chairman* of Testing Division
Lyman C. Aldrich, West Virginia Pulp & Paper Co.

Lyman Aldrich stated that he had been appointed acting chairman by W. K. Wilson, chairman, since none of the regular members were able to attend. John Cousins, chairman of the Pulp Testing Committee, was present and assisted Mr. Aldrich in explaining to visitors present the rights and responsibilities of the committee and the testing division organization. An invitation was given to all visitors to join the committee and help in the fields of their particular interest. Notes on the committee meeting in New York at the annual meeting were read to the committee.

T 428 "Water-Soluble Acidity or Alkalinity of Paper" and T 435 "Hydrogen Ion Concentration (pH) of Paper Extracts" were first discussed. John Henderson stated the hot extraction did not give true results and others agreed. This agrees with opinions previously expressed at other meetings. Dr. Clark stated the boiling method should be maintained because it is largely used for aging characteristics. Ed Brandon stated many government specifications include hot water extraction. Mr. Aldrich stated that it was undesirable to have two versions in the same method which give different results. It was decided to appoint. John Henderson, Crown Zellerbach, to a subcommittee with Sterling Bailey of the North Carolina Pulp Co. with the assignment of rewriting these methods. These methods, to be regrouped, and renumbered, include the following:

Cold extraction, determine both pH (with a pH meter) and acidity or alkalinity.
Hot extraction, determine both pH (with a pH meter) and acidity or alkalinity.

The revision of T 19 "Pentosans in Wood" and T 223 "Pentosans in Paper" were next considered. The presentation of W. K. Wilson's paper on comparison of the aniline acetate, orcinol, and bromination methods by Dr. Hobbs at the conference was noted. Mr. Aldrich reviewed the history of these revisions, stating that an approved revision had been withdrawn because of the inaccuracy found by Wilson in the aniline acetate method. There was only moderate interest in pentosan determinations shown. Cousins suggested the revision be left with current subcommittee members since they were competent and experienced.

Mr. Aldrich stated that a new method "Kappa Number of Pulp" to replace the present T 214 "Permanganate Number of Pulp" and the revision of T 202 "Chlorine Consumption of Pulp" were being reviewed by the Standards Committee and would soon be published.

Bruce Chamberlin asked if the committee was considering analysis of sulfite in NSSC black liquor. Several stated this was normally run by acidifying the black liquor and using the procedure for sulfite in sulfite black liquor. The writing of a method was tabled until the next meeting at which time it should be determined if there is other interest in such a method and if it warrants "Standard" classification.

The meeting was adjourned at 3:30 p.m.

W. K. Wilson, *Chairman*

Fourdrinier Committee

The fall meeting of the TAPPI Fourdrinier Committee was held at the Towne House Motor Inn, Rochester, N. Y., on Monday, Oct. 26, 1959. Chairman Kennedy called the meeting to order at 10:00 a.m. The following were in attendance:

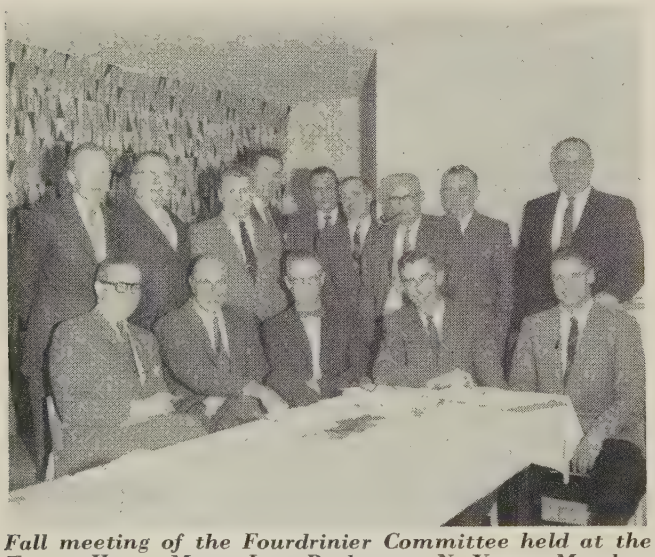
C. L. Brown
R. Fraser
J. Hannegan
P. H. Hershey
W. H. Kennedy
R. N. King
S. J. Leishman
A. E. Levensailor
R. C. Masterman
H. H. Morton
H. H. Mullins
R. W. Peters
J. E. Schad
A. J. Winchester

The minutes of the committee meeting of May 4, 1959, were read and approved.

On Project No. 635, "The Problems of High-Speed Machine Operation," Mr. Levensailor reported on the results of the tabulation of the questionnaire. He felt that there was such a variety of grades that the results were inconclusive except perhaps that all have pressure headboxes and are not running up to design speed. Mr. Winchester said that even though the results were inconclusive we had an obligation to those returning the questionnaire and, therefore, a special report should be submitted. To this end Mr. Levensailor will draw what conclusions he can, submitting a summary report and a suggested outline for tabulation to TAPPI Headquarters who will publish this report according to the instructions from Mr. Levensailor.

A discussion of the results indicated that the scope of the questionnaire was possibly too broad. Mr. Fraser reported that a similar survey of the Canadian newsprint industry also was inconclusive but that he would send Mr. Levensailor a copy of the report as a possible format. It is possible that certain segments of the questionnaire are worthy of further investigation and Mr. Mullins and Mr. Brown will assist Mr. Levensailor in further analysis of the questionnaire to determine whether subjects of more limited scope should be enlarged on.

On the "Wet End Process Control" project (no project number assigned as yet) Mr. Hershey reviewed the letter of transmittal and the questions on a proposed questionnaire.



Fall meeting of the Fourdrinier Committee held at the Towne House Motor Inn, Rochester, N. Y., on Monday, Oct. 26, 1959. Standing (left to right): P. H. Hershey, P. H. Glatfelter Co., Spring Grove, Pa.; H. H. Mullins, Olin Mathieson Chemical Corp., W. Monroe, La.; A. E. Levensailor, International Paper Co., Mobile, Ala.; R. N. King, Nash Engineering Co., S. Norwalk, Conn.; C. L. Brown, Crossett Paper Mills, Crossett, Ark.; R. Fraser, Manitoba Paper Co., Pine Falls, Me.; S. J. Leishman, Provincial Paper Co., Ltd., Thorold, Ont.; H. H. Morton, Paper Mill Superintendent, Cumberl and Mills, Md.; J. Hannigan, International Paper Co., Corinth, N. Y. Seated (left to right): A. J. Winchester, TAPPI, New York, N. Y.; R. W. Peters, Eastman Kodak Co., Rochester, N. Y.; R. C. Masterman, International Paper Co., Glens Falls, N. Y.; W. H. Kennedy, The Black Clawson Co., Watertown, N. Y.; J. E. Schad, Consolidated Water Power & Paper Co., Wisconsin Rapids, Wis.

The committee felt the scope of the questionnaire was too broad and redefined the limits as "stock preparation to reel." The questionnaire was referred back to the subcommittee of Messrs. Hershey, Leishman, and Spencer and Messrs. Kennedy and Peters were added to the subcommittee. The subcommittee will make an effort to rework the questionnaire according to the reduced scope and report at the next committee meeting in February.

The meeting was adjourned for lunch and reconvened at 2:15 p.m. At this time Chairman Kennedy opened a planned "bull session" by going around the table and asking each member to pose a question for comments by the rest of the committee. Such subjects as use of Teflon suction-box covers, vacuum differential in suction box, synthetic drier felts, Extremultus belt drive, moisture control, and calender stack crowning were discussed. This took a longer time than Chairman Kennedy had planned but was so interesting and successful that the committee decided to have a similar session in February.

Project No. 734, "Dandy Roll Practices" was opened for discussion. Mr. Hannegan reported that a tentative questionnaire had been submitted to Johnston, Sinclair, Cheney Bigelow and Plank companies for comment. The questionnaire was also reviewed by the committee and it was decided that Messrs. Hannegan and Schad should put the questionnaire in final form and submit a covering letter and the questionnaire to Headquarters office for approval and issuance to all fourdrinier mills. It was decided that before the questionnaire was submitted to mills that Messrs. Hannegan, Schad, and Winchester should draft a letter to be sent for a preliminary survey as to their willingness to answer the questionnaire and the number of questionnaire forms they desire to cover their various machines or grades. It is hoped that the survey letter would be ready by December 1, the questionnaire submitted by January 1, and a preliminary tabulation of the results reported at the February meeting. A final report would be submitted at the May meeting.

On Project No. 737, "Rope Carrier Practices," Mr. Mullins reported on his work as to the origin and development of rope carriers. Mr. Mullins read a preliminary draft of a paper on the subject and it was decided that he should put the finishing touches to the paper and submit it to TAPPI Headquarters for approval. It will be delivered at the Fourdrinier Committee general session in February.

At this point there was a discussion as to the optimum committee size. Mr. Winchester felt that it was not too large yet and that there is no optimum size, but he felt that a committee should not be so large that every member on it does not have a job to do.

Mr. King was asked to explore the possibility of embarking on a new project dealing with variations in vacuum across the suction box of a suction roll. He is to report at the February meeting on the work to date and possible techniques in furthering this investigation.

A new project which might be entitled "Suction Box Covers" was discussed and assigned to Messrs. Fraser and Morton. It was suggested that this could very well develop into a full-fledged research project and that we may wish to request a research appropriation. Messrs. Morton and Fraser are to discuss the project and outline the scope and objectives. Mr. Winchester will forward a blank Committee Project Proposal Sheet to Mr. Morton so that a project number can be assigned. It is hoped that enough information will be accumulated on the possibility of conducting semicommercial experiments at the University of Maine and to the various types of suction box material available so that a decision can be made at the February meeting.

The Fourdrinier Committee is tentatively planning on three activities at the February meeting in New York. The regular committee meeting will be held on Monday afternoon, the technical session will be held probably on Wednes-

day morning if this does not conflict with the Cylinder Committee session and a third get-together of the committee for a planned "bull session."

The committee meeting was adjourned at 5:30 p.m.

On Tuesday morning those of the committee who could remain in Rochester overnight visited the Kodak Park plant of the Eastman Kodak Co., for a bus trip around the plant and a tour through the Eastman Kodak Co. Paper Mills. The meeting was concluded with a luncheon in the Kodak Park Dining Room.

R. W. Peters, *Vice-Chairman*

Container Testing Committee

The meeting was called to order at 9:00 a.m. by Chairman, M. J. Clark. The following members were present:

M. J. Clark, *chairman*, Inland Container
R. W. Hoffman, *vice-chairman*, Continental Can Co.
G. C. Lecky, *secretary*, St. Regis Paper Co., F. J. Kress Box Div.
B. Q. Haynes, B. F. Perkins & Son Co.
H. B. Johnson, International Paper Co.
E. A. Knapp, West Virginia Pulp & Paper Co.
W. B. Lincoln, Jr., Inland Container Corp.
C. S. Macnair, Acme Steel Co.
T. J. Muldoon, Fibre Box Association
W. S. McDonald, International Paper Co.
H. G. Nelson, Owens-Illinois Glass Co.
H. J. Ostrowski, Hinde & Dauch Co.—Canada
H. W. Seibel, Gaylord Container Corp.
H. J. Schlotter, Hoerner Boxes, Inc.
R. E. Swingle, Acme Steel Co.
L. J. Trimer, Mead Containers
A. D. Wolvin, Weyerhaeuser Timber Co.
C. J. Zusi, Consultant

Visitors:

R. L. Beach, Jr., International Paper Co.
W. Lindsey, Olin Mathieson Chemical Corp.
J. R. Little, West Virginia Pulp & Paper Co.—H & D Div.
D. M. Long, River Raisin Paper Co.
L. A. Shipman, Olin Mathieson Chemical Corp.
O. P. Wanamaker, Fiberboard Paper Products Co.
J. E. Wright, Stone Container Corp.
A. J. Winchester, TAPPI

D. M. Long requested he be assigned to the committee.

The minutes of the February meeting were approved as published. The secretary reported that the committee had been circularized to find out what members were still interested in remaining on the committee. With very few exceptions, all members wished to retain their present status and the current committee membership is as listed in the 1959 TAPPI Yearbook.

Subcommittee Reports

Flat Crush Subcommittee. The chairman of the Flat Crush Subcommittee, Mr. Nelson, passed out sheets showing the significance of the speed of loading, type of loading and specimen size on the flat crush, as well as a summary sheet on the Analysis of Variance for the Flat Crush Study. A discussion followed on the statistical study which showed in general that:

1. The results obtained on the National Forge Machine were significantly lower than those obtained on the H & D.
2. The rate of loading had a singificant effect on the flat crush results, with results tending to be higher as the rate of loading was increased.
3. Sample size and shape has very little effect on the test results, with the square-cut samples showing slightly lower readings than the round, and the 5-sq. in. slightly higher then the 10-sq. in.

In a discussion of the loading rate, it was brought out that the loading rate is the load rate of platen to platen, not the actual load rate on the sample which would be affected by the sample height and would not be a constant. It was decided in the new procedures to specify that the load rate is a platen to platen and not a sample load rate.

The chairman asked Hugh Johnson to draft a flat crush procedure to be submitted to Mr. Nelson and then to the committee for discussion, to be completed for our February meeting. This procedure was to follow standard TAPPI rules for test procedure and he could call on Jack Winchester for guidance and advice as to correct format. It was suggested that it be written up with the beam-type tester specified, a loading rate of 25 lb per sec. and a 10 sq. in., round sample.

The chairman thanked Mr. Nelson for the time and hard work he had spent on this project and for his very complete and well thought out presentation.

Mullen Test Subcommittee. As the Mullen Test Subcommittee chairman was not present, Mr. Knapp led the discussion regarding the Mullen test procedure. The first thing discussed was the changes made in the diaphragm and diaphragm plate of the Mullen tester.

Mr. Haynes of B. F. Perkins stated that the only specifications they are bound by are those contained in Rule 41, but that they, as manufacturers, make no changes unless they are requested to by customers. The changes in the diaphragm were made because of complaints of the short life of the old diaphragm, and after a subcommittee of the Container Testing Committee had requested that they improve the diaphragm. The new diaphragms have a mold number on each diaphragm and have been supplied on all orders for about a year. The new diaphragm holder with the rings on it instead of the bead is only being supplied to customers who specifically request it. B. F. Perkins has drawn up a standard procedure for testing the diaphragm distention.

A letter to the committee from F. D. Long was read in which he charged that the diaphragm and diaphragm plate change was tending to lower Mullen tests and to the paper suppliers this means extra expense to bring up their Mullen. It was the consensus of those of the committee who had used the new diaphragm that it was not causing lower Mullen tests, but that official approval should be given only after official study by the committee.

In the discussion it was brought out that top plate warp affects the Mullen test and is sometime more a factor than the diaphragm.

It was decided that the committee would make no recommendations at the present time but would appoint a task force to study the effect of the new plate and diaphragm on the Mullen. They would also try out the new ribbed diaphragm. This task force would be headed by Earl Knapp and he would set up a series of Round Robin tests using two types of diaphragms and two types of plates to determine whether any difference exists in using the different top plates and diaphragms. Mr. Haynes said B. F. Perkins could furnish the new plates.

Those who were appointed to participate in the Round Robin were:

Earl Knapp, *chairman*
R. W. Siebel
G. G. Maltenfort
R. C. McKee
C. H. Root
J. J. Kipnees
H. G. Nelson
H. J. Clark
G. C. Lecky

These tests would be made of liner and combined board. It is hoped they would be completed by January 1, 1960, so results could be tabulated by the February meeting.

Compression Test Subcommittee. The Compression Test Subcommittee chairman, Ked Martin, could not be present because of other committee meetings, and discussion of progress by this committee was postponed until the next meeting.

Other Activities. ASTM Committee D-6 is now balloting on a new procedure for the Concora Test. When this is approved by them, it will be brought up before our committee to see if it is acceptable for a TAPPI Standard.

The chairman reviewed his status report on projects that are the responsibility of the Container Testing Committee. A list of these projects appears on page 27 of the 1959–60 TAPPI Yearbook under Authorized Committee Projects. On Project No. 71—Revision of the Drop Test, it was decided to rewrite this test to conform to the ASTM test and recirculate it by mail for ballot.

The next meeting will be in conjunction with the National Meeting in February in New York.

The meeting adjourned at 12:15 p.m.

M. J. Clark, *Chairman*
G. C. Lecky, *Secretary*

Members of the Drying Committee at the 14th Engineering Conference, Pittsburgh, Pa.

Drying Committee

The meeting of the Drying Committee was called to order by S. G. Holt, chairman, at 12:15 p.m. on Oct. 12, 1959, at the Penn Sheraton Hotel, Pittsburgh, Pa. The minutes of the previous meeting were approved.

Members in attendance:

S. G. Holt, Consolidated Water Power & Paper Co. *chairman*
K. K. Khandelwal, Dewey & Almy Chemical Division, W. R. Grace & Co., *secretary*
A. J. Cirrito, Rice Barton Corp.
R. M. Cook, Ross Midwest Fulton Corp.
E. G. Drew, Drew Engineering Co.
L. G. Janett, J. O. Ross Engineering Corp.
N. R. Reitzel, West Virginia Pulp & Paper Co.
R. C. Swaney, S. Morgan Smith Co. & Beloit Iron Works
L. R. Vianey, The Eaton-Dikeman Co.
S. C. Williams, Dominion Engineering Works, Ltd.

New members in attendance:

G. Kerikorian, Rice Barton Corp.
R. C. Portouw, Drew Engineering Co.

Guests in attendance:

I. W. Edwards, Dominion Engineering Works Ltd.,
K. H. Jones, J. O. Ross Engineering Corp.
E. J. Justus, Beloit Iron Works
D. M. McCutcheon, Westinghouse Bettis Atomic Power
V. P. Owens, Combustion Engineering, Inc.
H. L. Smith, Jr., Horace L. Smith, Jr., Inc.
J. M. Temphill, Singmaster & Breyer, Inc.
A. J. Winchester, TAPPI

Members absent:

L. Blomgren
C. H. Cash
F. L. Cessna
H. P. Fishwick
W. A. Green, Jr.
A. Jallencreutz
S. T. Han
G. E. Jackson, *vice-chairman*
J. J. Killian
G. R. Lydick
J. M. Mallory
W. F. Overly
R. A. Paramore
F. G. Perry
H. W. Stevens
R. E. White

General

D. M. McCutcheon, author, and guests were introduced.

Mr. Holt indicated that all committee members had shown interest in subcommittees, and that they should be active on one or more subcommittees and projects.

The committee will hold two interim meetings a year besides the meetings at the February New York TAPPI Conference and the annual Engineering TAPPI Conference.

The interim meeting of the Drying Committee was held at the Airport Hotel at the Pittsburgh, Pa., airport on Dec. 8, 1959.

Future effort should be to set up good active projects which will lead to papers for presentation.

Status of Active Committee Projects

Project No. 505—Drying Rates of High Pressure Dryers: Subcommittee member, L. G. Janett, reported that the questionnaires brought about good results. To date, responses were received from 60 Yankee machines and about 150 fourdrinier machines. Next phase of subcommittee activity will be to correlate statistical data and write a paper from this survey. It was mentioned that this survey should be a good jumping-off point to high velocity air driers evaluation.

Project No. 750: This project was established to prepare a paper on wet pressing. R. E. White's paper, presented the following day terminated this project.

Project No. 755—Data Sheets Subcommittee: CPPA Data Sheets—Mr. Holt reported that Subcommittee Chairman H. W. Stevens has been communicating with CPPA, and a report should be available at the next interim committee meeting. Mr. Holt will write to Mr. Stevens on this subject.

Project No. 756—Drying Fundamentals Subcommittee: L. R. Vianey, member of the subcommittee, has contacted Gerber (Louisville) and General Electric Co., both manufacturers of high-frequency current generating equipment. Both these organizations had not done much work on use of high-frequency current for paper drying. Mr. Vianey will recommend the future direction of this project to his subcommittee.

Project No. 758: A paper on the Flakt drier will be presented at this convention; the project will then be closed. Owing to an impending strike Mr. G. E. Jackson was absent, and a replacement from his organization will present the paper.

*Project No. 759—Gas Turbines in Pulp and Paper Mills—interim report:** Mr. Swaney reported on this project on the use of gas turbines in paper mills. The following interim report was submitted.

Objective of project was to investigate the applicability of gas turbines to pulp and paper mill machinery for drives and waste heat recovery.

For the purpose of this project, a size range of 300 to 3000 hp. was selected as being most likely to be applicable to pulp and paper mill operations. An article entitled "Gas Turbine Progress," appearing in the November, 1958, issue of *Mechanical Engineering,* included a table listing the foremost manufacturers of gas turbines and indicated the range of sizes each manufacture, and many other classified specifications.

The results of this project are not encouraging. It was

hoped this project would reveal some interesting advantages to the pulp and paper industry and lead to some advancement and development of a profitable nature. Such is not the case.

While there are hundreds of successful installations of gas turbines throughout the world, they are all justified because of a peculiar problem or problems such as the luck of any other source of mechanical energy, unlimited waste fuel, or extremely remote location. Gas turbines seem very adaptable to installation in electric generating stations for carrying peak loads frequently and intermittently because the gas turbine can be put on the line and develop peak output from a cold start in a matter of a very few minutes; also, it can be retired instantly. In such cases, intermittent peak load availability offsets adverse economics.

Letters were written to General Electric, Westinghouse, Brown-Boveri, and Clark Brothers from the above-mentioned article and to the Elliott Co., and four replies were received. Also, a visit was made to Allis-Chalmers and a discussion with R. C. Allen, director of mechanical engineering, proved most helpful, even though Allis-Chalmers has temporarily abandoned their gas turbine development in favor of more pressing steam turbine development. Of the manufacturers contacted, only one can offer one size gas turbine for industrial duty such as in pulp and papermaking; it is 1220 hp.

The stumbling block is economics. The gas turbine is still relatively inefficient as well as vulnerable. Efficiencies of approximately 12% are not unusual. Gas turbines are vulnerable to contaminants in the fuels, and all residual fuels must be treated chemically, which seems to be both expensive and chemically variable. Natural gas, if in abundance, seems to be the most suitable and cheapest fuel, but unless there are some unusual circumstances, the gas turbine cannot compete with other energy generating plants. In every instance, the waste heat must be utilized to the utmost, and this is often complicated. Economic heat balances are almost impossible to maintain. However, an application such as ship propulsion presents a special problem that involves other factors which are offset by the gas turbine disadvantages. The classic statement in all the data obtained was from one manufacturer who suggests using the exhaust gases from the gas turbine at temperatures of about 800°F., for combustion of air in steam generating boilers, because the exhaust gases from the turbine contain about 16% free oxygen. Since air contains 23.2% free oxygen, it is quite obvious how inefficient gas turbines are.

If other than natural gas is used for fuel, the chemical treatment of the fuel would likely preclude using the exhaust gases for drying, and they could not be used directly. They would first have to go through an intermediate heat exchanger or heat exchange process to reduce their temperature to safe drying temperatures.

The most encouraging aspect of this project is the knowledge that the gas turbine manufacturers use large quantities of high grade paper on which to print their data and elaborate brochures. Truly, they are both voluminous and genuine works of art.

Project No. 760—Air Jet Driers: The purpose of this project is to obtain an engineering and economic analysis of high velocity air jet driers.

Dr. Claes Allander, Research Manager of AB Svenska Flaktfabriken, Stockholm, Sweden, will be invited to present a paper on air drying.

E. J. Justus of Beloit Iron Works has been invited to present a paper on air drying.

The main purpose of this subcommittee is still not achieved as it involves finding a speaker who is not connected with any of the suppliers of air driers.

Mr. Jannett proposed that another subcommittee be established to propose a testing procedure for air driers as a guide post so that sensible comparisons could be made of all kinds of air driers in the market. The air-drying field is new and not enough field data are yet available.

Papers for the 1960 Jacksonville meeting of the Engineering Division: "Infrared Drying" by H. L. Smith, Jr., and a paper by W. L. Ingenanson.

Lars Blomgren has translated the Swedish paper on hot press and has the approval of the author. He is to send this paper to H. O. Teeple who will have it published in *Tappi*. A new Drying Committee project number should be given to this paper.

Committee Membership

G. Keriforian of Rice Barton Corp. and R. C. Portouw of Drew Engineering Co. are to submit applications for committee membership.

Mr. Justus of Beloit Iron Works will request one of his people to join the committee.

Subcommittee Memberships

No change. A questionnaire is to be sent to all the Drying Committee members with regard to their subcommittee interests.

It was proposed that the Subcommittee on Radiant Heat Drying be reorganized into two subcommittees as follows: All Radiant Heating Methods and Electrical Drying Methods Such As Conductive and Dielectric Heating.

The meeting was adjourned at 2:45 p.m.

K. K. Khandelwal, *Secretary*

TAPPI Drying Committee Members

S. G. Holt, Consolidated Water Power & Paper Co., Wisconsin Rapids, *chairman*
G. E. Jackson, Bowaters Southern Paper Corp., Catawba, S. C., *vice-chairman*
Krishna K. Khandelwal, Dewey & Almy Chemical Division, W. R. Grace & Co., Cambridge, Mass., *secretary*
L. Blomgren, American SF Products, Inc., New York, N. Y.
G. H. Cash, Darien, Conn.
F. L. Cessna, Jr., Sorg Paper Co., Middletown, Ohio
A. J. Cirrito, Rice Barton Corp., Worcester, Mass.
R. M. Cook, Ross Midwest Fulton Corp., Dayton, Ohio
E. G. Drew, Drew Engineering Co., Portland, Ore.
H. P. Fishwick, Mason-Neilan Regulator Co., Norwood, Mass.
W. A. Green, Jr., Manchester Board & Paper Co., Richmond, Va.
A. Hallencreutz, American SF Products, Inc., New York, N. Y.
S. T. Han, The Institute of Paper Chemistry, Appleton, Wis.
L. G. Janett, J. O. Ross Engineering Corp., Chicago, Ill.
J. J. Kilian, West Virginia Pulp & Paper Co., Research Center, Covington, Va.
G. Kerikorian, Rice Barton Corp., Worcester, Mass.
G. R. Lydick, The Beckett Paper Co., Hamilton, Ohio
J. M. Mallory, Union Bag-Camp Paper Corp., Savannah, Ga.
W. F. Overly, Overly's, Inc., Neenah, Wis.
R. A. Paramore, Central Fibre Products Co., Hutchinson, Kan.
F. G. Perry, Arthur D. Little, Inc., Cambridge, Mass.
N. M. Reitzel, West Virginia Pulp & Paper Co., Covington, Va.
R. C. Portouw, Drew Engineering Co., Portland, Ore.
H. W. Stevens, S. D. Warren Co., Cumberland Mills, Me.
R. C. Swaney, Carlisle, Pa.
L. R. Vianey, The Eaton-Dikeman Co., Mt. Holly Springs, Pa.
R. E. White, Villanova University, Villanova, Pa.
S. G. Williams, Dominion Engineering Works Ltd., Montreal, P. Q., Canada

Routine Control

The annual fall meeting of the Routine Control Methods Committee was called to order by Chairman J. R. Gunning at 12:15 p.m. in the Studio Room of the Hotel Multonomah,

* Submitted by R. C. Casper Swaney and F. L. Cessna.

Portland, Ore., Aug. 18, 1959. The following members and guests were present:

J. R. Gunning, Abitibi-Provincial Paper Co.
R. S. Hunter, Hunter Associates Lab.
A. J. Winchester, TAPPI
James d'A Clark, Consulting Engineer
Ralph E. Green, Thwing-Albert Instrument Co.
Leon M. Tyler, Howard Paper Mills, Inc.
Lyman C. Aldrich, West Virginia Pulp & Paper Co.
Wm. H. Aiken, Diamond Gardner Corp.
M. P. Boland, Columbia-Southern Chemical Corp.
D. Noel Obenshain, West Virginia Pulp & Paper Co.
B. Q. Haynes, B. F. Perkins & Son, Inc.

There were no representatives of firms in the Northwestern area present.

Secretary Haynes read the minutes of the February 24 New York meeting. R. S. Hunter moved that they be accepted as read. L. C. Aldrich seconded the motion. They were unanimously accepted as read.

Mr. Clark brought up the subject of the manner in which Routine Control Methods were being published. He expressed some doubt on the advisability of publishing the names of the manufacturers of the equipment involved. It was argued that the equipment used must be identified by the name of its manufacturer in order to avoid confusion and improper use of the method. It was recommended that a better way would be to keep the method as general as possible and publish the name of the manufacturer only in instances where competing makes of the same type of apparatus could not qualify for the method as outlined.

Secretary Haynes then read a letter dated July 10, 1959, from D. J. MacLaurin of the Institute of Paper Chemistry resigning from the Routine Control Methods Committee. Before taking action on his resignation Dick Hunter pointed out that the Institute should be represented in some manner on the Routine Control Methods Committee. It was the general feeling of the committee that Mr. MacLaurin's inability to take an active part in the committee because of other duties was not sufficient cause for him to feel that he should resign. Dick Hunter suggested, and it was generally agreed, that Chairman Gunning take action on the resignation by sending a letter to Mr. MacLaurin requesting him to remain on the committee as a representative of the Institute of Paper Chemistry.

Mr. Gunning then raised the question of why Routine Control Methods were not published regularly in *Tappi*. Jack Winchester replied that it was a make-up room problem common to all publications. Articles were selected for publication according to contents, length, etc., to fill the space available. The solution to the problem was to create a backlog of methods from which the make-up room could always draw material to fill the available space.

Chairman Gunning then read a letter from C. L. Stumpp dated July 21, 1959, stating that because of other duties he would be unable to continue editing the Routine Control Methods and thought it best to resign as chairman of the editorial committee.

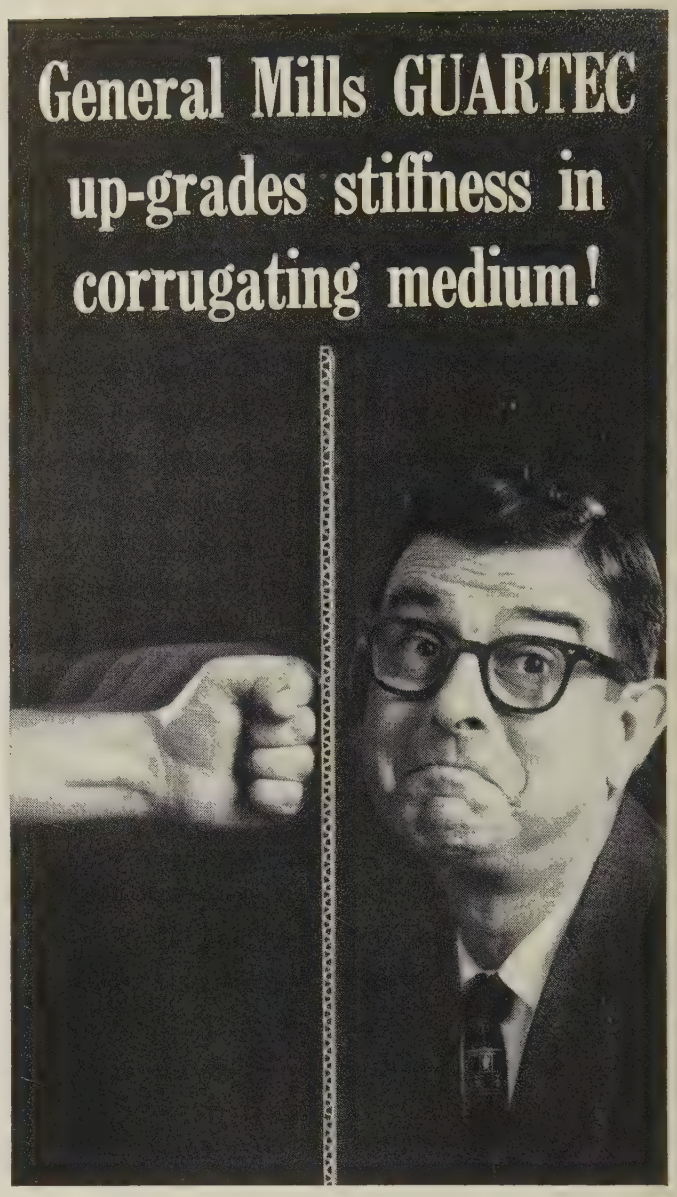
Lyman Aldrich volunteered to discuss the problem with Mr. Stumpp and see what could be done about editing and releasing the methods now under review. It was the general feeling that methods should be edited prior to publication if possible, but not at the expense of withholding publication of useful and necessary methods.

A method entitled "A Coating Pick Test Using the Interfiber Bond Tester" was submitted to Jack Winchester for publication.

J. R. Gunning read a letter from the Packaging Institute requesting permission to use the Routine Control Methods indexing system. Following a discussion, it was agreed that Chairman Gunning grant them permission to use the system.

The meeting was adjourned by Chairman Gunning at 1:55 p.m.

B. Q. HAYNES, *Secretary*



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ENGINEERING DIVISION

Questions and Answers

Good engineering questions are solicited as well as answers or alternate answers to the published questions. Material should be sent to TAPPI Headquarters, marked for the attention of H. O. Teeple. The items contributed will be forwarded to the Question and Answer Committee for processing. If comments are to be contributed, please refer to the question number.

(46) *Question:* **Excitation for synchronous jordan and refiner motors in our mill is distributed from a single 125-v. distribution bus. The main bus in our switchboard is the standard positive, negative, and equalizing arrangement and all bus bars are of the same cross section. Two 60-kw. compound-wound interpole generators are arranged for parallel operation and are properly connected through two three-pole switches to the switchboards. Field rheostats have been provided for each machine. The generators are almost flat compounded i.e., there is only about 2 v. rise from no load to full load. We have never been able to satisfactorily parallel these generators. No matter how carefully we balance the voltages when a second generator is connected, one or the other of the generators will motor and the other will take all of the load. Our load has grown so that it will overload one generator. How can we parallel these generators?**

(*46.1*) *Answer:* It is very difficult to parallel compound-wound generators when they have a rising or even a flat characteristic. It will be necessary for you to obtain a slightly drooping characteristic on each machine, i.e., about 5 v. less at full load than at no load. I suggest that a German silver shunt be prepared and connected across the series field of each generator until the proper characteristics have been obtained. This connection is necessary in order to weaken the series field effect. The operation should not take too long even if it is of the trial and error process.

*(47) *Question* (**Conversion of Baumé Degrees to Specific Gravity**): **How is the specific gravity of a liquid estimated from the degrees Baumé?**

*(*47.1*) *Answer:* The following formulas are used:

For liquids lighter than water—

$$\text{Sp. gr.} = \frac{140}{130 + \text{deg. Bé.}}$$

For liquids heavier than water—

$$\text{Sp. gr.} = \frac{145}{145 - \text{deg. Bé.}}$$

*(48) *Question* (**Wattmeters Require Adjustment**): **On one of the feeders going out from our plant the voltage is stepped up from 2300 to 13,200 and, at the end of a 5-mile transmission line, stepped down again to 2300 v. Power is metered on the 2300-v. side of the transformers at both ends of the line. Should not both meters register the same? As it is now, the meter at the out end of the line registers considerably more than the one at the power plant.**

*(*48.1*) *Answer:* The only case where the accuracy of electrical measuring instruments is affected by a change in the length of the leads is when the instrument is connected to a shunt, such as d.c. ammeters and in some cases the current coils of wattmeters. With alternating-current instruments connected to the secondaries of transformers the length of the leads may be changed without affecting the accuracy of the instrument.

*(49) *Question* (**Reduction of Baumé Reading to Pounds of Oil per Gallon**): **Knowing the Baumé reading of a fuel oil or distillate, how are the pounds per gallon estimated?**

*(*49.1*) *Answer:* The weight per gallon will be equal to the weight of a gallon of pure water multiplied by the specific gravity or weight of the oil as compared with the weight of an equal volume of pure water. One U. S. gallon of pure water at 62°F. weighs 8.3356 lb. and 1 Imp. gal. of pure water at the same temperature weighs 10 lb. To convert degrees Baumé to specific gravity, the following formulas are used:

For liquids heavier than water, sp. gr. = 145 ÷ (145 − deg. Bé.)

For liquids lighter than water, sp. gr. = 140 ÷ (130 + deg. Bé.)

Hence for an oil lighter than water, if the reading per the Baumé hydrometer is 21° Bé. the specific gravity would be 140 ÷ (130 + 21° Bé) = 0.927, and the weight would be 8.3356 × 0.927 = 7.727 lb. per U. S. gallon.

*(50) *Question* (**Laying Up Steam Turbines**): **We have two impulse turbines which may be operated condensing or noncondensing. Having arranged to operate our plant by electric power received from a public central station, what should be done in laying up the turbines to stand by in good condition, ready to start when needed?**

*(*50.1*) *Answer:* When the turbines are shut down and drained, pour about ½ gal. of engine oil in the cylinder of each turbine, when slowly turning over, and work the oil through the cylinder to coat thoroughly the blading, packing glands, and other finished parts. Care should be taken that the turbines are completely disconnected from any live or exhaust steam. To guard against rust or damage from such connections, close the throttle valves and provide bleeders outside of the valves with independent openings to the atmosphere. Any drains connected with other drips should be disconnected close to the machines, and the drain pipes should be blanked off to prevent steam from backing up to the turbines. After

Note: Questions and answers marked with asterisk (*) are reproduced courtesy *Power* magazine.

the turbines are thoroughly drained, close the exhaust valves and open a bleeder outside of the exhaust valves to prevent any steam backing up from other connections. After all finished parts have received a doping of thick oil, provide the turbines with tarpaulins or canvas coverings to protect them from dust.

*(51) *Question* (**Voltage Drop in Field Winding and Rheostat**): **In a d.c. generator how does voltage drop in the field rheostat compare with that in the field winding?**

*(*51.1*) *Answer:* If the generator is shunt-wound, it is customary to design the field rheostat to absorb about half of the line voltage, while in compound-wound generators this is usually reduced to about one third of line voltage. The voltage drop in the rheostat will therefore be equal to that in the field winding in the first case and to half of the drop in the field in the second case.

*(52) *Question* (**Oil Grooves**): **Is oiling aided by grooves in bearings?**

*(*52.1*) *Answer:* General opinion is that oiling is improved, providing the grooves are properly designed and cut. They should not be too deep or they will break the oil film, nor too shallow, or they fail of their purpose. They should not extend to the bearing edge, or will act as drains, and should not be cut in the lower bearing. Oil should be admitted at a minimum pressure point if possible.

*(53) *Question* (**Moisture in Transformer Oil**): **What is a simple method of testing transformer oil for moisture?**

*(*53.1*) *Answer:* Anhydrous copper sulphate when placed in transformer oil will give a bluish tint if moisture is present. If small pieces of hot iron or nails are dropped into a sample of the oil the presence of moisture will be indicated by a hissing sound. Before applying either of these tests, it will be advisable to make the tests on samples known to contain moisture and others that are known to be "dry," to become familiar with reactions under both conditions.

*(56) *Question* (**Leveling and Landing**): **What is the difference between an automatic-leveling and an automatic-landing type elevator?**

*(*56.1*) *Answer:* An automatic-leveling elevator will automatically level into a floor when making a stop. Assume that the car when on up motion stopped 3 in. above the floor. The control is so arranged that the car will automatically start down and come level with the landing. The automatic-landing type elevator stops the car level with the floor. If the control gets out of adjustment and the car stops 3 in. away from the floor, the control will not level it into the landing. The trouble can only be corrected by adjusting the control.

*(57) *Question* (**Causes of Turbine Vibration**): **What causes a turbine to vibrate?**

*(*57.1*) *Answer:* Unbalance and misalignment are usually the causes of the few cases of vibration that appear. Water coming over with the steam will cause multistage turbines to vibrate but usually does not affect single-stage or small machines. Electrical causes which throw a strain on the revolving electrical element cause it to be pulled out of line and produce vibration sometimes. Other causes of vibration are loosening, warping, or failure of parts of the rotor or stator, eccentric or loose coupling, spring in shaft, settling of the foundation, uneven expansion, sympathetic vibration, or vibration which is easily set up in the foundation, floor, piping, or other parts connected with the turbine; strains caused by exhaust piping of steam piping, excessive variation of vacuum in some cases, the restriction of oil or cooling water; improper action of the valve gear. Lack of accurate alignment is the principal cause of turbine troubles, and greatest care in adjustment is required. The ordinary test for first-class operation is to balance a coin on the bearing bolts. If this will stand satisfactorily on edge, operation is considered good. Small flexible parts, such as brackets, piping, etc., may vibrate considerably although general operation is good.

Unanswered Questions

(36) *Question:* **Calculating pipe friction for stock pump sizing has always been more of a guess than a science because so little is actually known about the behavior of the stock slurries. The recent work by the University of Maine has shown how conservative all the known U. S. data has been and the test results are as comprehensive as can be devised with any test setup. So that data on stock pipe friction could be collected on a grand scale, would it be worth while to the industry to organize as many pulp and paper mills that would cooperate in such a venture and have them perform at least one pipe friction test on their own particular pulp. This would only require two pressure gage connections in any straight run or irregular pipe line and a consistency sample taken during the test. The data could be tabulated by TAPPI and the results published in a new set of data sheets.**

(37) *Question:* **Some years ago the TAPPI Electrical Engineering Committee published a report on cable practices and applications for the pulp and paper industry. This has some good dope in it, but now it's a little out of date. When are you going to bring it up to date?**

(38) *Question:* **We hear and read a lot about computers these days. Please tell us whether or not it makes sense for a large mill to have one. What kind of problems would we use it for and what about the economies?**

(39) *Question:* **At 125 lb. steam pressure, the temperature of saturated steam is 352.9°. This steam has 1192.2 B.t.u. total heat. The latent heat of vaporization of water to steam is 956.7 B.t.u. If 100°F. superheat is added, the temperature of the steam then becomes 452.9°. Is the drying rated on paper driers (pounds of steam per pound of paper) increased through the use of this superheat?**

(40) *Question:* **Is any pulp mill using a chip analyzer based on Canadian Standard D-18 (TAPPI Standard _____ measures chip size but not chip length)?**

(41) *Question:* **What, if any, defects in hard concrete surfacing for log yard water flume aprons are appearing after extensive use?**

(42) *Question:* **In vertical pumps of the turbine, mixed, or axial flow type, where bearings are lubricated by the liquid pumped, what bearing materials and life have been experienced by the pulp and paper industry? What are the pump applications?**

(43) *Question:* **What materials are most successful in submerged bearings, flooded by pulp stock, as in the case of stock chest agitators? Is external purging with fresh water found generally practical?**

Qualitative Analysis of Mineral Filler and Mineral Coating of Paper

Proposed Revision of T 421 m-44

(THIS STANDARD IS UNDER THE JURISDICTION OF THE CHEMICAL METHODS COMMITTEE)

THIS method is for the qualitative analysis of mineral constituents of filled and coated papers. The presence of such mineral matter is indicated by the amount and appearance of the ash. If the ash content is not much over 1% and the ash is light and fluffy in character, added mineral filler is probably absent. If more than 1%, and the ash is dense and compact, added filler is likely, unless waste papers have been used in the furnish; this at times may be determined microscopically (see TAPPI Standard T 401 m).

The procedure described is generally satisfactory for determining the kind or kinds of filler used, but occasionally in a borderline case, a quantitative analysis may be necessary. The presence of some fillers may be determined directly by microscopic analysis (see TAPPI Standard T 488 sm).

In order to interpret qualitative ash tests with greater assurance, it is important to estimate the relative amounts of the various constituents found. In this connection, for example, the light voluminous character of an aluminum hydroxide precipitate should be taken into account when relating its quantity to that of a heavy, dense precipitate like barium sulfate. With flame and colorimetric tests, it is particularly difficult to judge the relative quantities of the constituents present and these should be interpreted with special care.

The percentage of ash present may be determined in accordance with TAPPI Standard T 413 m, and the residue so obtained used for analysis.

REAGENTS

Use distilled water and analytical grade chemicals.

A. *Hydrochloric Acid*, conc. HCl (sp. gr. 1.19); also approximately 2 *N* (15 ml. conc. HCl diluted to 100 ml.).

B. *Sulfuric Acid*, conc. H_2SO_4; also approximately 5% (3 ml. conc. H_2SO_4 diluted to 100 ml.).

C. *Cobalt Nitrate Solution, 5%*. Dissolve 8 g. of $Co(NO_3)_2 \cdot 6H_2O$ in 100 ml. of water.

D. *Hydrogen Peroxide*, 30% H_2O_2, or a solution of 3% U.S.P. H_2O_2 used in proportionately greater quantities.

E. Lead Acetate Paper. Immerse strips of filter paper in a saturated solution of $Pb(C_2H_3O_2) \cdot 3H_2O$.

F. *Lime Water*, saturated solution. Dissolve about 0.2 g. of $Ca(OH)_2$ in 100 ml. of water and filter.

G. *Magnesium Reagent.* Dissolve 0.5 g. of *p*-nitrobenzeneazoresorcinol in 100 ml. of 1% NaOH solution.

H. *Microcosmic Salt Solution.* Dissolve 5 g. of $NaNH_4HPO_4$ in water and dilute to 100 ml.

I. *Ammonium Sulfate*, $(NH_4)_2SO_4$, crystals.

J. *Sodium Carbonate*, Na_2CO_3, powdered.

K. *Other Reagents* (see TAPPI Standard T 610 m): acetic acid, CH_3COOH (sp. gr. 1.05); ammonia, conc. NH_4OH; ammonium chloride, 10% solution NH_4Cl; ammonium oxalate, 3.5% solution $(NH_4)_2C_2O_4 \cdot H_2O$; barium chloride, 10% solution $BaCl_2 \cdot 2H_2O$; potassium dichromate, 4% solution $K_2Cr_2O_7$; potassium ferrocyanide, 1.5% solution $K_4Fe(CN)_6 \cdot 3H_2O$; sodium hydroxide, 10% solution NaOH.

APPARATUS

1. *Platinum Crucible.*
2. *Charcoal Block and Blowpipe.*
3. *Platinum Wire Needle.*

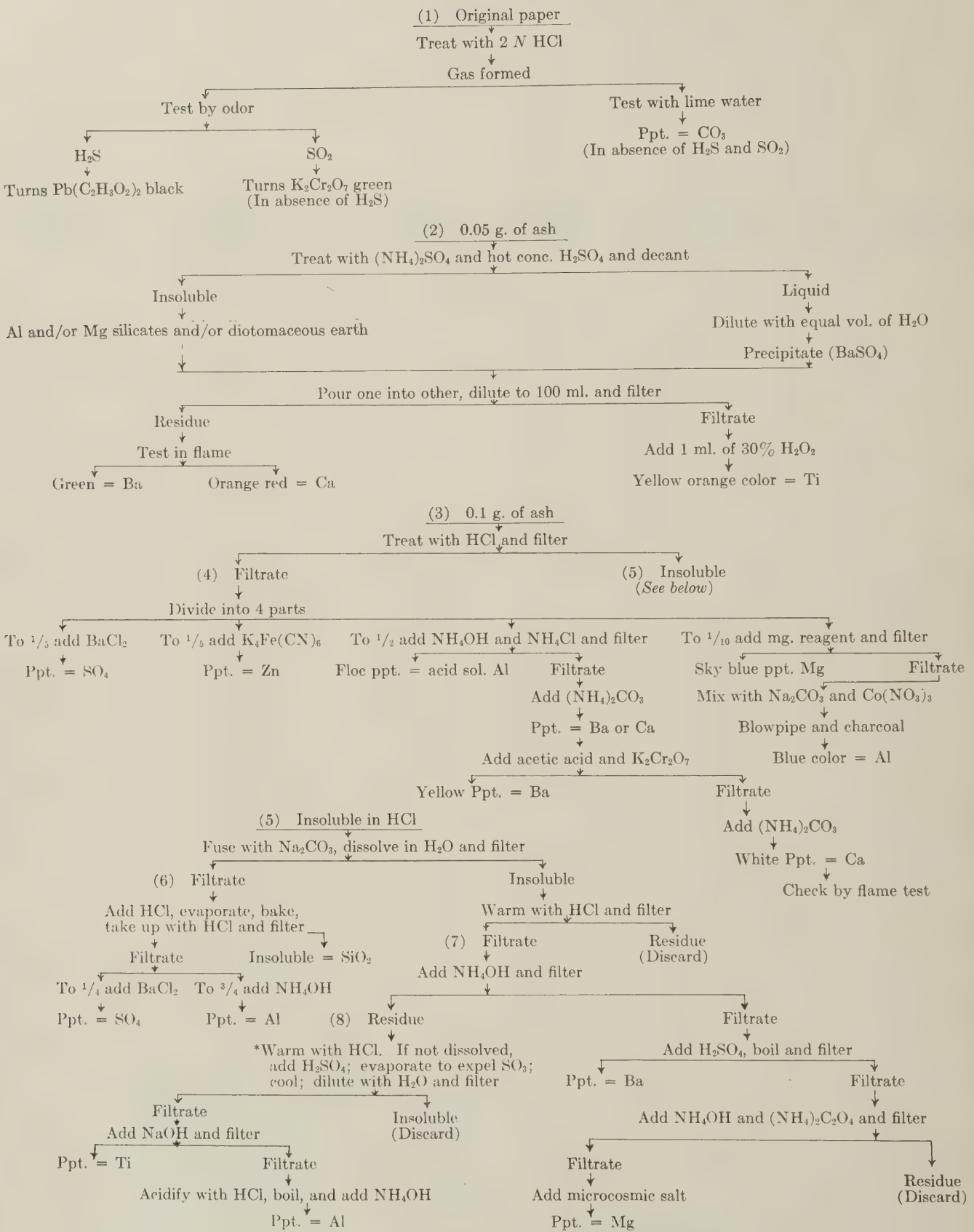
TEST SPECIMEN

Select sufficient paper which is representative of the sample to yield approximately 0.15 g. of ash. Ash at a temperature of about 600°C. (lower than in TAPPI Standard T 413 m). Retain portions of the original paper for testing for sulfites, sulfides, and carbonates since these are altered in composition by ashing. If the paper is coated and separate analyses of filling materials and coating minerals are desired, remove the coating by the procedure described in TAPPI Standard T 407 m. Evaporate the aqueous mixture containing the coating minerals to dryness and ash the residue. Also ash the base stock and analyze these two portions separately.

Note: If synthetic coating adhesives are used in place of starch or casein, enzymatic stripping will not work. In this case, scrape the coating off the surface with a razor blade.

PROCEDURE

An outline scheme of the complete procedure is given in Fig. 1.



* This procedure is necessary only if both Ti and HCl-insoluble Al compounds are present and it is necessary to estimate relative amounts of Ti and Al.

Fig. 1. Qualitative analysis of mineral coating and filler

(1) Sulfite, Sulfide and Carbonate

Treat a portion of the unignited coating or paper sample in a small beaker or a test tube with 2 *N* HCl. Note whether effervescence takes place and the odor, if any, of the escaping gas. SO_2 and H_2S indicate sulfites and sulfides, respectively. Warm the contents of the beaker and test the vapor with moistened lead acetate paper. The development of a metallic gray or black color confirms the presence of sulfide. In the absence of sulfides, add either a small crystal of potassium dichromate or a few drops of a 4% solution to a small portion of the HCl solution. A green coloration indicates a reducing agent; in this case probably a sulfite.

Note: As far as is known, mixtures of sulfites and sulfides are not used in loading or coating paper.

If sulfites and sulfides are absent, effervescence alone is a good indication of the presence of a carbonate, which may be confirmed by holding a glass rod with a drop of saturated lime water just above the solution. A cloudy precipitate indicates the presence of CO_2. This precipitate may later dissolve. A confirmatory test for CO_2 in the presence of sulfites is to add to the contents of the beaker, a weak solution of iodine (about 0.1 *N*) drop by drop until the entire liquid is colored yellow to oxidize the sulfites to sulfates. Then test with lime water on a glass rod as described.

(2) Al or Mg Silicates, Ca or Ba Sulfates, TiO_2

To approximately 0.05 g. of ash add 10 g. of $(NH_4)_2SO_4$ and 20 ml. of conc. H_2SO_4. Cover with a watch glass and boil vigorously for at least 3 min.

Considerable undissolved matter indicates the presence of aluminum and/or magnesium silicates and/or diatomaceous earth. If the hot solution is clear, aluminum or magnesium silicates and diatomaceous earth are absent. (Calcium and barium sulfates will be dissolved unless the sample being specimen so tested weighs much more than 0.05 g.)

Decant some of the liquid portion, cool, and dilute it cautiously with a portion (up to about five times its volume) of cold water. The formation of a precipitate on dilution, indicates the presence of barium sulfate, which is relatively soluble in hot conc. H_2SO_4.

Mix the diluted mixture with the remainder of the undiluted mixture from the conc. H_2SO_4 treatment and add sufficient water to make the ratio of H_2O to H_2SO_4 about 5 to 1. (If the original H_2SO_4 solution was clear, dilute it 5 to 1 by adding water cautiously after cooling.) Filter out any residue and to the cooled solution add 1 ml. of 30% H_2O_2. A deep yellow or orange color indicates the presence of titanium, the depth of color being proportional to the amount present. If only a very light yellow color is produced, it may be caused by TiO_2 from clay, if present, or derived from TiO_2 in the mill water.

The presence of barium or calcium in any insoluble residue can be determined by a flame test. Filter the solution, wash the residue with 5% H_2SO_4, dip a clean platinum wire into the moist residue, and hold it in a Bunsen flame. A green flame indicates the presence of barium, a red flame indicates calcium, and a yellow to colorless flame, indicates aluminum or magnesium silicates, or both.

Note: $CaSO_4$ is quite soluble in dil. H_2SO_4 and may not be observed at this step.

(3) Sulfide, Sulfite, and Carbonate

Treat 0.1 g. of the ash with 10 ml. of water and 5 ml. of conc. HCl. Effervescence, as with step (1), indicates the presence of a carbonate or sulfite. Withdraw 1 or 2 ml. of the solution and add two drops of 4% $K_2Cr_2O_7$ solution. A green coloration indicates sulfite.

Heat the contents of the beaker to boiling and test the fumes with moistened lead acetate paper; the development of a metallic gray or black color indicates the presence of sulfides. These tests should be checked by tests made on the original specimen of paper since carbonates may be lost, sulfates may be reduced to sulfites or sulfides or sulfites oxidized to sulfates, depending on the temperature and oxidizing conditions during ignition.

Note: If the temperature of ashing is over 900°C. as specified in TAPPI Standard T 413 m, no carbonates will be present and any sulfites probably would be oxidized to sulfates.

Boil the mixture 5 min. or longer unless complete solution occurs sooner. Add 35 ml. of water, and again heat to boiling. If this solution is not clear, filter and wash twice with water, reserving the filtrate for analysis of the acid-soluble portion. Wash the insoluble residue thoroughly and discard the washings. Reserve the acid-insoluble portion for later treatments (4) and (5).

(4) Sulfate, Zn, Mg, Al. Ba, Ca

To one-fifth of the acid-soluble portion from (3) add 1 ml. of $BaCl_2$ solution. A precipitate which appears immediately or after heating for 10 min., shows the presence of sulfates. To another one-fifth portion add a few ml. of potassium ferrocyanide solution. A heavy white precipitate indicates the presence of zinc. The presence of zinc may be confirmed by the red color produced in the caustic soda-dithizone test (see Feigl: Spot Tests).

To a small portion of the other three-fifths of the solution (about one-tenth of the total) add one or two drops of the magnesium reagent and make alkaline with NaOH solution; a sky-blue precipitate indicates the presence of magnesium but all other minerals commonly used in paper give a violet coloration to the solution. Take care not to add an excess of the reagent, since it is liable to mask the blue precipitate. If in doubt, filter the solution and examine the filter paper for the presence of a blue precipitate.

Mix the solution with an excess of Na_2CO_3 on a charcoal block, and moisten with a very small amount of $Co(NO_3)_2$ solution. A permanent blue coloration upon heating with a blowpipe flame confirms the presence of aluminum. (Take care not to mask the blue coloration by using an excess of cobalt nitrate solution.)

To the remainder of the solution add an excess of NH_4OH and NH_4Cl solutions. Aluminum will appear as a white floc if it is present in acid-soluble form. Filter and add $(NH_4)_2CO_3$ solution to the filtrate. A white precipitate indicates the presence of barium and calcium. Without filtering, make the solution acid

with acetic acid (which dissolves the precipitate) and add $K_2Cr_2O_7$ solution. If barium is present in an acid-soluble form, a yellow precipitate will show its presence. Filter and add an excess of $(NH_4)_2CO_3$ solution. Since barium is removed by the preceding step, a white precipitate indicates calcium. Check the precipitate for calcium by a flame test.

(5) Silicate, Sulfate, Al

Place the unfolded filter paper containing the remainder of the acid-insoluble portion from (3) in a platinum crucible. Dry and ignite with free access of air until all organic matter is removed. Add 1 to 2 g. of Na_2CO_3 and fuse until a clear melt is obtained or until all reaction has ceased. Decompose the melt in 25 ml. of hot water, heat to boiling, filter, and wash the paper thoroughly with water. Reserve the filtrate and the first two washings for the analyses of the water-soluble portion for silicate and aluminum.

Wash the insoluble portion from the filter paper back into the original beaker with a stream of water, place the beaker under the funnel, and pour 10 ml. of hot conc. HCl through the filter paper. Reserve the contents of the beaker for analysis of the water-insoluble portion according to section (6).

Make the water-soluble portion slightly acid with HCl, evaporate to dryness in the platinum dish, and bake at 100 to 105°C. for 0.5 to 1 hr. Moisten the residue with conc. HCl, let stand a few minutes, then add 5 to 10 ml. of dil. HCl, and warm. A light, flocculent, insoluble residue in the solution indicates the presence of silicate which is best observed by viewing against a dark background. Filter, and to one-fourth of the filtrate add $BaCl_2$ solution; a precipitate indicates sulfates. To the remainder of the filtrate add NH_4OH. A precipitate indicates aluminum.

(6) Al, Fe, Ba, Mg

Warm the acid solution of the water-insoluble portion from (5), until no more material dissolves, add 50 ml. of water, boil, and add a very slight excess of NH_4OH to precipitate aluminum (and iron). Filter and wash; reserve the precipitate for analysis according to (7) below. To the filtrate add 5 ml. of 5% H_2SO_4 to precipitate barium, boil, and filter. Make the filtrate ammoniacal, add a little ammonium oxalate solution to insure the absence of calcium, filtering if any precipitate appears; then add to the cold solution 5 ml. of NH_4OH, an excess of microcosmic salt solution and stir well. A precipitate appearing in 15 min. will indicate the presence of magnesium compounds.

(7) Ti and Al

The following procedure is necessary only if both titanium and HCl-insoluble aluminum compounds are present and it is desired to estimate the relative proportions of titanium and aluminum. Wash the insoluble precipitate obtained by addition of NH_4OH in (6) with water, and then transfer it to the original beaker with a stream of water. Add 5 ml. of conc. HCl and warm. If solution is not complete, add 3 ml. of conc. H_2SO_4 and heat until solution occurs, then drive off the SO_3 fumes over a free flame under a hood. Cool and dilute to 35 ml. Any $BaSO_4$ not decomposed by the Na_2CO_3 fusion will be insoluble at this point and should be removed by filtration. Make the solution neutral with 10% NaOH and add an additional volume equal to that of the neutralized solution. Heat to boiling, cool, and filter. Titanium hydroxide remains insoluble. Neutralize the filtrate with HCl, heat to boiling, and make slightly ammoniacal. If any precipitate forms, it is $Al(OH)_3$.

REPORT

Report all cations, anions, and radicals found, and indicate their relative amounts present such as trace, slight amount, considerable, large amount, etc. It is desirable also to interpret and report results of the analysis in terms of the fillers or mineral coating materials indicated to be present.

INTERPRETATION OF RESULTS

For the fillers listed, positive tests will be obtained in the procedural sections indicated. See also Additional Information.

Calcium carbonate—CO_2 (1), Ca (4).
Calcium carbonate with magnesium hydroxide or carbonate—CO_2 (1), Ca (4), Mg (4).
Calcium sulfate—Ca (4), SO_4 (4).
Calcium sulfite—SO_2 (1), Ca (4).
Barium carbonate—CO_2 (1), Ba (4).
Barium sulfate—Ba (6), SO_4 (5).
Lithopone—Sulfide (1), Zn (4), Ba (6), SO_4 (5).
Zinc oxide—Zn (4).
Zinc sulfide—Sulfide (1), Zn (4).
Titanium dioxide—Ti (2), or (7).
Titanium dioxide-barium sulfate—Ti (2) or (7), Ba (6), SO_4 (5).
Titanium dioxide-calcium sulfate—Ti (2) or (7), Ca (4), SO_4 (4).
Satin white (coating)—CO_2 (1), Al (4), Ca (4), SO_4 (4).
Clay—Al (5) and (7), SiO_2 (5).
Tale or asbestine—Mg (6), SiO_2 (5).
Diatomaceous earth—SiO_2 (5).

ADDITIONAL INFORMATION

The analysis can be considerably simplified if it is desired only to establish the presence or absence of a particular filler.

The use of sulfides as filling or coating materials in the paper industry is restricted to zinc sulfide alone or in combination with $BaSO_4$ (lithopone). A positive test for zinc in the absence of sulfide indicates the use of zinc oxide in the paper. A combination of the oxide and sulfide cannot be identified as such. The amount of zinc pigments in paper can be determined by TAPPI Standard T 438 m. The use of sulfites is restricted to calcium sulfite.

Most commercial fillers contain impurities which may lead to incorrect conclusions if only small amounts or traces of constituents are found. For example, satin white may contain carbonate; clays, especially domestic clays, contain a small quantity of titanium and clays may also contain calcium and magnesium; titanium dioxide may contain small amounts of aluminum and sulfate; calcium fillers may contain magnesium; sulfide and sulfite fillers usually contain sulfates.

The common use of alum in papermaking leads to the presence of aluminum compounds in appreciable quantities, even when fillers are absent. Small quantities or traces of calcium, magnesium sulfates, etc. are observed in many papers containing no filler and are derived from the mineral constituents of the pulp

or left in the paper before drying, particularly from a mill in which the water is hard.

Carbonates together with considerable acid-soluble calcium indicate the presence of calcium carbonate which may exist as chalk or whiting. If HCl-soluble magnesium is also present, a mixture of calcium and magnesium carbonates is indicated. A combination of barium and carbonate may exist as witherite. Barium in this form will be shown in the HCl-treated portion of the ash since barium sulfate is insoluble in dilute HCl.

A positive test for acid-soluble sulfates and calcium indicates the use of calcium sulfate as crown filler, gypsum, or satin white, etc. If considerable HCl-soluble aluminum is also present in the coating, the mineral used may be satin white. If carbonates are used in conjunction with sulfides, there may be a positive test for sulfates when none are present.

A positive test for calcium and sulfite indicates the presence of calcium sulfite.

The presence of considerable magnesium and silicate indicates the use of talc, agalite, or asbestine. Silica may also indicate the presence of diatomaceous earth. The characteristic diatom forms may be readily recognized on microscopic examination.

The residue from the portion of the ash treated with concentrated H_2SO_4 may be clay, talc, diatomaceous earth, or a mixture of these substances. A positive test for aluminum indicates that clay was used.

Barium sulfate (barytes or blanc fixe) is indicated by the formation of a precipitate on the dilution of the H_2SO_4 solution and by a positive flame test for barium on the residue.

Titanium may be present as titanium dioxide alone or mixed with barium or calcium sulfates. Titanium-barium mixtures are not likely to be found in paper made in North America, but titanium-calcium mixtures may be found in papers made in America and abroad.

Any of these may be used in conjunction with other fillers—for example, with clay. The quantitative determination of titanium pigments in paper is given in TAPPI Standard T 439 m.

A microscopic examination of the ash (see TAPPI Standard T 488 sm) usually proves to be a useful adjunct to chemical analysis, and if possible should be attempted.

The main changes of this revision are in section (4) of the procedure. Other changes are editorial.

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Apparatus

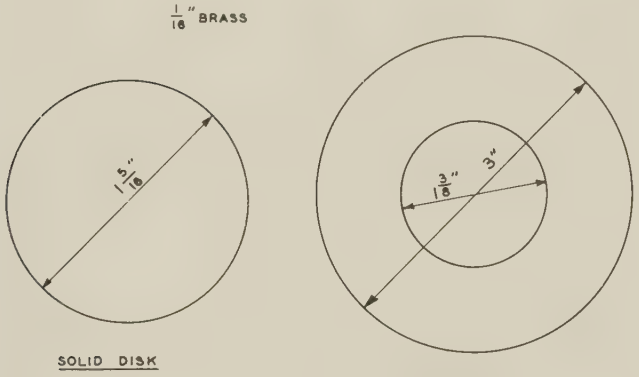
Jumbo Mullen Tester

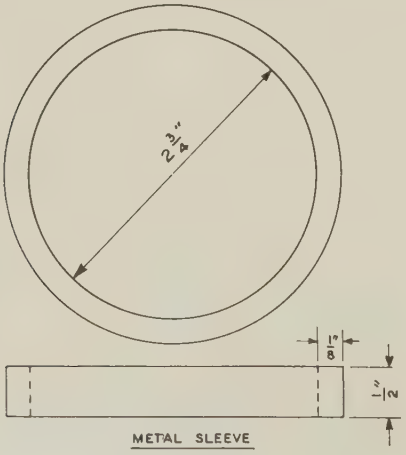
Brass disks, $^1/_{16}$ in. thick and $2^5/_{16}$ in. diameter

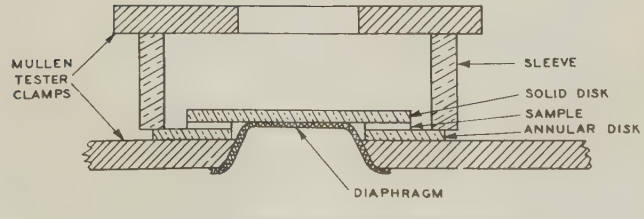
Annular brass disks $^1/_{16}$ in. thick, 3 in. outer diameter and $1^3/_8$ in. inner diameter

Steel sleeve about $2^3/_4$ in. inside diameter, $^1/_2$ in. high and $^1/_8$ in. thick

Means of cleanly cutting an annular sample of $2^5/_{16}$ in. outer diameter and $1^3/_8$ in. inner diameter







CROSS SECTION OF SAMPLE-AND-DISK ASSEMBLY DURING TEST

Procedure

Cut a sample of the board to be tested, 14 in. long by 3 in. wide. Cover each side with a strip of 3-in. double-face pressure sensitive tape without pealing the protective liner. From this strip cut four annular specimens for testing. Peel one of the protective liners from each sample and press lightly to one of the solid disks; then peel the other liner and place an annular disk on the other side, using the hole in each for alignment.

Press the sample between the disks under about 100 p.s.i. A convenient means of doing this is the sample clamp of the Mullen tester itself. A pile of a dozen samples may be pressed at one time.

Place one sample on the lower platen of the Mullen tester with the annular disk down and centrally located so that the hole in the disk is aligned with the hole in the platen. Place the steel sleeve upon the annular disk and clamp in place with the upper platen. Operate the tester until the expansion of the diaphragm against the solid disk ruptures the sample. Use the 0 to 200 p.s.i. gage.

Read the maximum pressure in the usual manner, and note the location of the rupture if this is of interest. Any indication of failure of the tape bond invalidates a test.

Since the area of contact between diaphragm and solid disk varies according to the pressure, do not calculate the pressure per square inch of sample, but report the results as gage readings, in pounds per square inch.

Contributed by: J. Donahue and W. Verseput, Continental Can Co., Inc. Robert Gair Paper Products Group, Uncasville, Conn.

This is a Routine Control Method and should not be considered as a Standard in any way. Corrections may be made before loose-leaf publication. Comments will be welcome and should be sent to A. J. Winchester, TAPPI, 155 E. 44th St., New York 17, N. Y.

RC 274. pH of Paper

This method is a quick control test for measuring the pH of paper by means of successive spots of buffered indicator solutions applied to the paper. It avoids hydrolysis and other effects sometimes encountered in normal extraction procedures.

Materials

Buffered indicator solutions covering the desired pH range. Unbuffered indicator solution. Figure 1 shows a convenient portable arrangement.

* *Note:* Solutions of the composition described can be purchased from the Hartman-Leddon Co., 5821 Market St., Philadelphia 39, Pa. These solutions will contain a biocide for their preservation against bacteria.

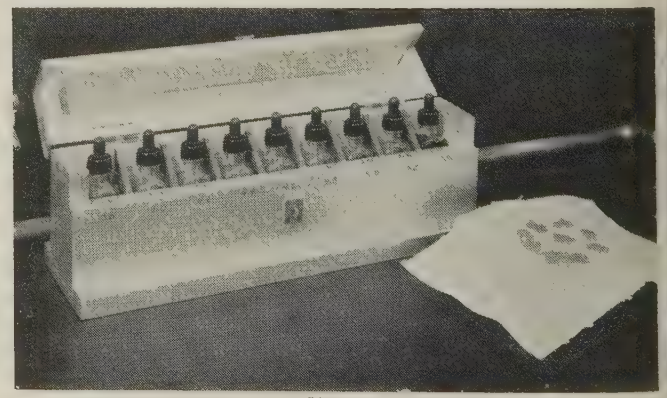


Fig. 1

Preparation of Indicator Solutions

(*a*) *For testing papers in the 3.8 to 5.4 pH range:* Weigh out 10.210 g. potassium acid phthalate and transfer quantitatively to a 1000-ml. volumetric flask. Add to the flask about 500 ml. distilled water and the amount (see Table I) of standard acid of alkali required to give the desired pH. Use a buret for this volume measurement. When all of the potassium acid phthalate ($KHC_6H_4O_4$) has dissolved, add the 40.0 ml. of 1% bromcresol blue (Table I) and dilute with distilled water to the 1000-ml. mark.

Table I. Quantities Required for One Liter of Indicator Solution Using 10.210 G. $KHC_6H_4O_4$ in Each

Desired pH	*To be added, ml.* 0.1000 *M HCL*[a]	0.1000 *M NaOH*[a]	1% *bromcresol green*	*Final volume, ml.*
3.8	26.00	...	40.0	1000
4.0	...	4.0	40.0	1000
4.2	...	37.0	40.0	1000
4.4	...	75.0	40.0	1000
4.6	...	121.5	40.0	1000
4.8	...	177.0	40.0	1000
5.0	...	238.5	40.0	1000
5.2	...	300	40.0	1000
Unbuffered[b]	...	...	40.0	1000

[a] If the standard acid and alkali on hand are not exactly 0.1000 molar, calculate and use the equivalent quantity of the available standard solutions.
[b] This solution is to contain no $KHC_6H_4O_4$. Adjust to 4.6–4.8 with minimum acid or base.

(*b*) *For testing papers in the 2.8 to 4.6 pH range:* Prepare suitable buffers using information available in any standard chemistry handbook. Use bromphenol blue as indicator.

(*c*) *For testing papers in the 5.4 to 7.0 pH* range: Prepare suitable buffers using information available in any standard chemistry handbook. Use bromcresol purple as indicator.

Test Procedure

Place a drop of unbuffered indicator solution on the paper sample. Around it, in a circle, place a drop of each of the buffered indicator solutions and note which color matches that of the unbuffered drop. This is the pH of the paper.

Notes and Explanation

The buffering action of these eight solutions is strong enough so that when a drop is placed on most papers the pH of the solution does not change and hence the color or shade of the drop will remain unchanged. The ninth bottle contains 0.04% bromcresol green adjusted to the neutral color and pH (4.6–4.8) but containing no buffer salts. Therefore, when this solution is placed on paper, it assumes the pH of the paper. This pH is determined by noting which drop of buffered solution on the paper matches the color of the unbuffered indicator solution.

If two or more of the buffered solutions turn the same color when dropped on a piece of paper, this indicates that the paper contains so much acid or alkali that it overpowers the buffer salts in the solutions. With such paper this method cannot be used. The color of some colored papers, especially the highly colored ones, obscures the color differences between the indicator spots and therefore makes the test less easy or impossible to carry out.

Submitted by JOHN W. EASTES, Research Associate, Chemical Research Division, Scott Paper Co., Chester, Pa.

This is a Routine Control Method and should not be considered as a Standard in any way. Corrections may be made before looseleaf publication. Comments will be welcomed and should be sent to A. J. Winchester, TAPPI, 360 Lexington Ave., New York 17, N. Y.

Eleventh Testing Conference
Pantlind Hotel, Grand Rapids, Mich.
Sept. 27–29, 1960

Get Rid of SCRAP and TRIM

...Quickly...Profitably

Insert shows how shredder is connected to one plant's present pneumatic waste pick-up system.

● Why waste valuable working space and create hazards hoarding reclaimable stock? Now it is easy to quickly shred all sizes and shapes of sheet corrugated or set-up corrugated boxes with an Industrial Heavy Duty Shredder. Will even convert double-double corrugated to random ¼" to 2" wide shreds ready for baling.

Baled paper is compact, easier, faster to handle. It takes up less than half the space of bundled stock, and can be sold instead of burning or hauling away. "Initial purchase and installation costs of the entire system will be regained in roughly one year thru reduction of labor in scrap handling."

These push-button controlled shredders can also be connected direct to pneumatic pickup systems or belt conveyors. Available in two sizes, 39½" throat and 58" throat. Both models are equipped with individual motor driven crushers which squash fully assembled boxes and shred them in one operation. Also available with single conveyor belt for shredding flat stock.

We'll gamble that an Industrial Shredder will add efficiency and improve house keeping in your plant. FULL SIZE DEMONSTRATOR AVAILABLE — MAKE RESERVATION FOR TRIAL USE.

INDUSTRIAL SHREDDER AND CUTTER COMPANY
128 MILL STREET • SALEM, OHIO

LOCAL SECTION ACTIVITIES

Meeting Calendar

January, 1960

21 Kalamazoo Valley Section, Harris Hotel, Kalamazoo, Mich., Papermakers' Get-Together.
21 Lake Erie Section, Cleveland Engineering and Scientific Center, Conference on Pulp and Paper Testing Equipment and Methods Including Laboratory Coating and Sheetmaking Techniques.
21 Empire State Section—Eastern District, Queensbury Hotel, Glens Falls, N. Y., Executives Night.
21 Delaware Valley Section, Engineers Club, Philadelphia, Pa., Operations Research Panel.
22–23 Southeastern Section, Jacksonville, Fla.
26 Pacific Section—Golden Gate District, visit to California Forest Products Laboratory, 3:30–4:00 p.m. Dinner at Hotel Claremont 6:00 p.m.
29 Gulf Coast Section, Stafford Hotel, Tuscaloosa, Ala.

February, 1960

2 Chicago Section, Chicago Bar Association, 29 S. LaSalle Street, Chicago, Ill., "Adhesives."
11 Ohio Section, Colony Restaurant, Swifton Center, Cincinnati, Ohio; plant tour through Formica, Div. of American Cyanamid.
11 Empire State Section—Northern District, "Color."
16 Ohio Section—Indiana District, plant tour to be announced.
18 Empire State Section—Eastern District, "Synthetic Fibers" (tentative).
18 Lake Erie Section, Colton Chemical Co., 6626 Union Ave., Cleveland, Ohio, plant tour.

March, 1960

1 Chicago Section, Chicago Bar Association, 29 S. LaSalle St., Chicago, Ill., "Folding Cartons."
8 Lakes States, mill visit to Bergstrom Paper Co., Neenah; dinner at Menasha Elks Club. Theme: "Statistical Methods Application in the Paper Industry."
8 Empire State Section—Metropolitan District, "Coating."
9 Empire State Section—Northern District, "Strength Development in Paper."
14 New England Section—Eastern District, Armand's Restaurant, Route 9, Framingham, Mass.
15 Ohio Section, Manchester Hotel, Middletown, Ohio, "Two-Phase Flow in a Wet Web," Speaker: Joe Parker, Beloit Iron Works.
15 Ohio Section—Indiana District, Marott Hotel, Indianapolis, Ind., Panel Discussion, to be announced.
17 Empire State Section—Eastern District, Junior Awards Contest.
17 Lake Erie Section, Cleveland Engineering and Scientific Center, "Off-Machine Coating with Emphasis on Waxing and Barrier Types and Base Paper Requirements."
17 Kalamazoo Valley Section, Inman's Restaurant, Galesburg, Mich., "Graphic Arts."
18 Gulf Coast Section, Cove Hotel, Panama City, Fla.
18 Virginia-Carolina Section, John Marshall Hotel, Richmond, Va.
18–19 Southeastern Section, Columbia, S. C.
26 Delaware Valley Section, Benjamin Franklin Hotel, Philadelphia, Pa., 1960 Phildelphia Graphic Arts Conference.
29 Golden Gate District—Pacific Section, "Recent Advances in Materials Handling," 4:00 p.m., mill visit to Nulaid plant. Dinner at Cottage Restaurant, San Leandro, 6:00 p.m.

April, 1960

5 Chicago Section, Chicago Bar Association, 29 S. LaSalle St., Chicago, Ill., "Corrugating."
8 New England Section, Management Night.
12 Ohio Section, Miami University, Oxford, Ohio, tour of the new paper school. Subject: "High Polymers," Speaker: H. E. Whiteacre.
12 Lakes States Section, dinner, location to be announced, C. J. West Awards.
12 Empire State Section—Metropolitan District, "Packaging."
14 Empire State Section—Northern District, Junior Award Night.
15 Pacific Section, Everett, Wash., Shibley Award Meeting.
19 Ohio Section—Indiana District, plant tour to be announced, "Coating." Speaker: Brag Golding, Purdue University.
21 Empire State Section—Eastern District, Symposium, "Wet End Machine Operation."
21 Kalamazoo Valley Section, Inman's Restaurant, Galesburg, Mich., "Stock Preparation."
28 Delaware Valley Section, Engineers Club, Philadelphia, Pa., E. J. Albert Award Program.

May, 1960

3 Chicago Section, Chicago Bar Association, 29 S. LaSalle St., Chicago, Ill.
5–7 Pacific Section, Bellingham, Wash., or Harrison Hot Springs, Annual Meeting.
10 Pacific Section—Golden Gate District, Hotel Claremont, Berkeley, Calif., True Memorial Lecture.
12 Kalamazoo Valley Section, Harris Hotel, Kalamazoo, Mich., Recognition Night, WMU Awards.
12 Empire State Section—Northern District, Annual Ladies' Night.
12 Ohio Section, Hartwell Country Club, Cincinnati, Ohio, Ladies Night.
13 Gulf Coast Section, Jung Hotel, New Orleans, La.
13 Empire State Section—Metropolitan District, Ladies' Night.
17 Ohio Section—Indiana District, Marott Hotel, Indianapolis, Ind., "Testing." Speaker: Arthur Dresfield, The Glidden Co.
20–21 Virginia-Carolina Section, Wilmington, N. C., mill visit to Riegel Paper Corp., Acme, N. C.
20–21 Southeastern Section, Savannah, Ga.
21 Empire State Section—Eastern District, Annual Ladies' Night.
27 Delaware Valley Section, Beloit Eastern, Downingtown, Pa., tour and outing.

June, 1960

3–4 New England Section, Chatham Bars Inn, Chatham, Mass., Spring Meeting.
10–11 Empire State Section, Whiteface Inn, Whiteface, N. Y., Annual Meeting.
17–18 Maine-New Hampshire Section, Mountain View House, Whitefield, N. H., Annual Meeting.
21 Kalamazoo Valley Section, Annual Golf Outing, Gull Lake Country Club.

September, 1960

To be announced.
Pacific Section, Salem, Ore., "Modernization."

Chicago Section

Following is a brief summary of the November meeting of the Chicago Section of TAPPI. The meeting was held November 2 at the Chicago Bar Association.

The subject of this second meeting of the season pertained to "New Trends in Corrugated Printing." Bernard Rataj, vice-president, Triangle Container Corp., was program chairman and arranged a panel of 3 speakers.

Don Ellinger, Downing Box Co., spoke on "The Dorr Rapi-Die Register System for Printer-Slotters."

Les Mendlin, Continental Can Co., covered "New Aspects in Corrugated."

Jim Carr, Greenwood Engineering Co., talked on the "Greenwood Five-Color Printer-Slotter."

The talks were well received by the more than 80 people in attendance. The talk by Mr. Ellinger covered a new method for rapid die mounting which assures accurate register and eliminates use of staples. Mr. Carr used slides in showing and discussing the basic principles of the Greenwood "Victory" printer-slotter.

A question-and-answer period concluded the evening's meeting.

M. H. Meyer

Delaware Valley Section

Philadelphia Graphic Arts Conference

The 1960 Philadelphia Conference of the Graphic Arts Industry will be held at the Benjamin Franklin Hotel in Philadelphia on Saturday, March 26, from 8 a.m. to 5 p.m.

This conference will commemorate the 270th anniversary of the founding of the paper industry in America by Wilhelm Rittenhaus and the 275th anniversary of the founding of the first print shop in the Middle Colonies by William Bradford.

This bigger and better Third Annual "Philadelphia Conference" is expected to establish the conference as an annual Philadelphia event growing larger each year and promoting greater mutual understanding and cooperation between papermakers and the graphic arts industry.

Preconference registration fee is at the bargain price of $7.50 versus $10 at the conference.

Send advance registration fee to Henry Smaine, P. H. Glatfelter Co., Spring Grove, Pa.

Planning for the Philadelphia Conference of the Graphic Arts Industry to be held at the Benjamin Franklin Hotel on March 26, 1960, are (l. to r.): G. Boger, chairman of the Delaware Valley Section of TAPPI; A. Moss, publicity chairman; Henry Smaine, TAPPI treasurer; A. Saindon, vice-chairman of the TAPPI section; Mr. Coyle, banquet manager of the hotel; H. Harcke of Graphic Arts, Inc.; Phil Cerasoli of TAPPI and Frederic Dannerth

The Albert Award

The Albert Award is an annual competition sponsored by the Delaware Valley Section of the Technical Association of the Pulp and Paper Industry.

This competition consists of the presentation of papers on original work of technical nature by young persons actively engaged in the paper industry, chemical suppliers to the pulp and paper industry, and pulp and paper mill equipment suppliers. It is considered that the monetary awards in themselves are secondary and merely a means of stimulating competition. The true value of the competition lies in the training, experience, and practice gained by the contestants in planning and executing the work, and in preparing and presenting the papers. These values are received, not alone by the winner, but are shared by every participant.

Qualification of Competitors: The Albert Award competition is open to every young person actively engaged in some phase of the pulp and paper industry, chemical suppliers to the pulp and paper industry or manufacturers of equipment for the pulp and paper industry. Since the primary purpose of the competition is to benefit the younger members of the industry, those in supervisory positions or those who have gained maturity through considerable experience, such as technical directors, chief chemists, experienced research chemists, district managers, supervisors and the like are not considered to be eligible participants. The executive committee of the section, however, has the authority to determine eligibility in individual cases.

Subject Matter for Papers: The participant may choose his own subject on some technical aspect of the manufacture of pulp and paper, and associated with some operation, process, or investigation in which he has been concerned in the course of his employment.

Preparation of Papers: Since it is the intention of the section to publish all suitable Albert Award papers in Tappi as well as other trade journals, it is suggested that the contestants follow the form outlined in the TAPPI Year Book in the section entitled, "Regulations Governing Manuscripts." In view of the projected publication of the papers, we request that clearance for such publication be obtained by each entrant, from his employer. However, such clearance, is not a prerequisite for entry. For those not having access to a TAPPI Year Book, the information may be obtained by contacting the Award Chairman, Joseph A. Paciello, E. I. du Pont de Nemours & Co., 1616 Walnut St., Philadelphia 3, Pa. The Award Chairman or the Section Secretary will be happy to supply any other information desired, as well.

The length of the Albert Award papers should not exceed, approximately, 20 min. presentation time.

Each contestant should submit to the Section Secretary, Thomas Czepiel, Scott Paper Co., Chester, Pa., *four* copies of his paper by the section meeting date, March 26, 1960. This will permit the judges to review adequately the paper prior to their presentation. It is desirable that each contestant declare his intention to participate in the competition, in writing, by March 1.

Papers to be eligible for the Award must be presented in person by the author at the regular meeting of the section,

Engineer's Club, 1317 Spruce St., Philadelphia, Thursday, April 28, 1960.

Judging of Papers: The papers will be judged by a committee of three, to be appointed by the section chairman prior to March 26, 1960. Papers will be judged on the following points: (1) Planning, resourcefulness, and originality exhibited in the execution of the work described. (2) Organization of the paper. (3) Presentation of the paper. These points of consideration are arranged in descending order of their weighting by the judges.

Nature of Award: The Albert Award shall consist of a first prize of $100, second prize of $50, and third prize of $25.

In addition, the winner will have his name engraved on a plaque to remain in his possession until the following year's competition. It is suggested that this plaque be displayed suitably and prominently at the winner's place of employment.

Each contestant, including the winner, shall receive a suitable token of his satisfactory participation, the nature of which shall be determined by the executive committee.

Announcement of the winner and presentation of the award shall be made at the end of the section meeting on April 28, 1960.

October Meeting

The Delaware Valley Section of TAPPI met for its monthly meeting on Oct. 22, 1959, at the Engineer's Club in Philadelphia. The feature presentation was a talk by Otto Berggren, field service engineer for the Huyck Felt Co., on the instruments developed by Huyck for measuring the running performance of felts.

Mr. Berggren received his degree from the paper school of the New York State College of Forestry and has worked closely with operating papermakers throughout the United States developing practical methods for improving felt performance and press section efficiency.

Mr. Berggren first discussed the application of the beta ray gage to the measurement of water removal in the press section of paper machines and related it to over-all felt-press efficiency. Actual mill results were used to explain in detail the application of this device.

The Huyck-Smyth porosity tester was next described as the only portable device available for the measurement of running felt porosity. Not only is this instrument capable of detecting filled up streaks in felts, but it also enables practical papermakers to predict to a high degree of accuracy the end point in the useful life of felts.

Mr. Berggren also described the electronic draw speed indicator which has proved very successful in tuning up all types of paper machines. This device is capable of giving instantaneous speed readings on the various machine components that enable the rapid detection of slippage and/or discontinuous loading. The Kenwood Felt measuring wheel, frequently used in conjunction with the electric draw speed indicator, was also explained as capable of providing felt stretch-curve measurements around the entire machine.

Field results obtained with the Huyck meter were also discussed. This device provides a quick and accurate means of determining actual felt tensions at various positions on the machine and offers a convenient method for pinpointing areas of excessive tension.

Otto Berggren of the Huyck Felt Co. speaking to the Delaware Valley Section of TAPPI at its October, 1959, meeting

Mr. Berggren's talk was concluded with a question-and-answer period.

Question (K. L. Fischer): Can felt life be improved by the use of continuous felt washers?
Answer: Generally yes, but how much felt life can be lengthened by continuous washing is difficult to predict.
Question (R. E. Rogers): Have you studied felt porosity as a function of running felt tension?
Answer: No, although work of this type is in progress.

New England (Eastern District)

The November meeting of the Eastern District of the New England Section of TAPPI was held on the ninth at Armand's Beacon Terrace in Framingham, Mass. There were 107 members present.

Edward J. Forbes, coordinator of corporate product planning for Itek Corp., spoke on "New Product Opportunities for Paper Makers and Coaters in Information Technology." An abstract of his talk follows:

Seated at the head table with the speaker were Roger Wiewel, chairman; John Wendell, vice-chairman; Philip Slayton, treasurer; Albert Lamoureux, secretary: Thomas Allan and Thomas Sargent of the Executive Committee, all of the Eastern District and John Lewis, chairman, and Alex Hutton, treasurer, both of the New England Section.

The next meeting of the New England Section will be on April 8, 1960, with Alfred Nissan of Rensselaer Polytechnic speaking on "Some Aspects of Hydrogen Bonding."

Mr. Forbes' talk was well received and was followed by a lively question-and-answer period exploring present opportunities, prospects, and limitations of the various systems of reproducing, storage, and retrieval of information.

The next meeting of the Eastern District of New England TAPPI was announced for Jan. 11, 1960, at Armand's Beacon Terrace with a speaker from Synco Resins talking on "Beater Additions of Phenolic Resins."

James G. Miller head of the Biology Dept. of Arthur D. Little, spoke at the September 14 meeting. Dr. Miller talked on "The New Food Additive Amendment to the Federal Food Drug and Cosmetic Act."

There were 116 present at the September meeting. Roger Wiewel was confined to his home with the grippe and asked John Wendell to preside. Whit Strecker spoke of our great loss and asked for a minute of silence out of respect for William T. Kirby, loyal and able treasurer of the New England Section.

A. C. Lamoreaux

New Product Opportunities for the Papermaker and Paper Coater—Abstract

Edward J. Forbes

The new graphic information handling area and Itek's general technical capabilities (optical, photographic, electronic, and mechanical) were briefly described: Itek-Boston being principally engaged in advanced development work in the reconnaissance and data reduction field: Photostat (Rochester) with the marketing of photostat papers and machines, and office copying, duplicating and microfilming equipment; Itek-Palo Alto with aero-space technology—particularly dynamic stability and thermodynamic analyses of space vehicles.

Itek is basically interested in the development of simple, low cost techniques for graphic information handling—particularly the storage and retrieval of information.

Edward J. Forbes, Corporate Product Planning, Itek Corp., Waltham, Mass.

Today, many basic paper products almost meet many needs in this area but the new high speed retrieval and print-out machines do seek new paper characteristics. We expect that paper will again prove its ability to give the customer adequate quality at the lowest possible price in direct competition with other possible image carrier materials.

New technology certainly is becoming increasingly important to paper manufacturers; for example, not many years ago photoconductive layers were laboratory curiosities prepared by evaporation and single crystal techniques. Now we find a negative-positive paper being used to make hard copy enlargements from selected microfilm images and paper companies are actively investigating the application of new photoconductive layers in water emulsion at a cost near that of the finish used in book and Bible papers.

General characteristics of this industry as reported in the last U. S. census of manufacturers show that in 1954 $11 billion of paper, pulp, and allied products were shipped, and of this $4.5 billion was value added by manufacture.

If sales of paper, pulp, and allied products are correlated with gross national product, it can be seen that since 1947 this industry has grown at a rate slightly greater than gross national product but net profit has dropped from 9.9% in 1947 to 4.8% in 1952—a much higher decrease in net profit than that experienced by all U. S. industry during the same period.

In fact, $562 million profit was earned on $5.7 billion sales in 1947 versus $408 million profit on $10.6 billion in sales in 1952.

These statistics suggest that the paper manufacturer should seek higher profit through higher productivity per employee. For some manufacturers this may well be achieved by producing a more costly product—e.g., coated papers—papers which would be very useful and economical for graphic information handling purposes.

Certainly modern papermaking techniques and technology involve a thorough understanding of the basic sciences, particularly physics and chemistry. This in turn leads to the development of all kinds of papers, films, foils, cloths, synthetic resins, and other components. If paper is examined as a base (or carrier) for a special coating, we find that there are today thousands of paper coatings and laminates which are merchandised through paper industry channels.

These coatings are sensitive to light, to a magnetic field, to current, and to pressure. Other useful coatings are abrasive, carbon, and stencil.

There are five basic types of coated paper that have special functional values: coated chart papers, magnetic papers, electrically conducting papers, plasticized papers, and photosensitive papers (e.g., blueprint and diazo). The methods of recording information on each of these papers were described, limitations noted, and some opportunities for improving the usefulness of some of these materials indicated.

Today coated chart papers (recording instruments) work effectively with a moving hot stylus pen or a beam of high intensity light. Tomorrow, graphic reproduction systems may be based on: corona discharge with electrostatic printing, or electrolytic or solid state chemical systems.

Magnetic paper tapes and sheets were introduced in the mid-1950's but displaced in the 1950's by stronger, higher priced, better quality magnetic coatings on plastic tape base. In the 1960's new graphic storage and print-out techniques may turn to cheaper papers where recording speeds of 8 in. per sec. for 6000 cycles is desired.

In spite of the fact that moisture is required for electrical current conduction, inert or reactive moving stylii systems offer interesting possibilities for new electrically conducting papers. With an inert stylus the point of contact current immediately makes a sharply defined stain with higher intensity achieved by higher voltage. The reactive stylus electrolytically plates or evaporates dark metal on white or translucent paper. This system may be limited by surface static or voltage.

The general competitive trend of the industry where better new products have replaced old established products is illustrated by for example, pressure-sensitive coated papers and films have largely replaced cloth-backed products; plastic magnetic tapes have replaced wire and metal for sound and instrument recording; more convenient Diazo papers have largely supplanted blueprint papers; conducting papers are beginning to show interesting applications in the facsimile and message field, and photoconducting papers eventually may compete with established photocopy papers.

It is concluded that opportunities do exist for the paper base and coating manufacturer to develop substantial new products especially for the graphic information handling field.

Golden Gate District

The December 1 meeting was held at Sabella's Capri Room on Fisherman's Wharf.

Approximately 75 men attended a most interesting panel discussion on Federal Food Additive Legislation at 3:30 p.m. Ralph W. Fogleman, manager of Hazelton Laboratories, Menlo Park, acted as moderator and the members of the panel were R. C. Nelson of the Legal Department of Crown Zellerbach Corp. and R. G. Mispley of the Manufacturing Department of Crown Zellerbach. The present position of the FDA was given by these men and a lively hour of questions and answers developed.

This was followed by a cocktail hour at 5:30 and then 95 men attended the dinner meeting.

Howard Baumgarten, corporate economist, Crown Zellerbach Corp., presented an excellent paper on "The Economics of the Paper Industry" which was enhanced by the use of color slides.

H. A. Harvey, *Secretary-Treasurer*

Kalamazoo Valley Section

The second meeting of the Kalamazoo Valley Section of TAPPI was held Nov. 12, 1959, at Inman's Restaurant, Galesburg, Mich. Despite inclement weather caused by the first heavy snow fall this winter more than 100 guests were present to hear John A. Sheridan of the La Salle Management Consultants, Inc., Chicago, Ill., discuss "The 1959 Labor Reform Law." The program chairman for the meeting was W. R. Noel.

Among the guests present were:

P. E. Nethercut, National TAPPI, Secretary-Treasurer
J. W. Field, National TAPPI, Local Section Liaison
H. O. Ware, National TAPPI, Executive Committee
V. E. Perry, Ohio Section, Chairman
C. E. Eberly, Indiana District (Ohio Section), Chairman
F. W. O'Neil, Empire State Section, Chairman
H. E. Pratt, Maine-New Hampshire Section, Secretary-Treasurer
H. J. Klauke, Chicago Section, Chairman
C. M. Helm, Chicago Section, 1st Vice-Chairman
R. R. Chase, Southeastern Section, Chairman
R. A. Flick, Southeastern Section, Vice-Chairman

Mr. Sheridan began his remarks by saying that his speech would deal with the most literal interpretations of the new labor law. He proceeded to outline a list of do's and don't's concerning management expenditures:

1. Management should not give any employee anything of value over and above his regular compensation since wrong circumstances may now make such a loan or gift illegal. Testimonial-type dinners for the purpose of persuading employees in their right to organize or not to organize also fall under this sanction.

2. Management should not pay any money to a labor organization, officer, or employee thereof.

3. Finally, management should not loan or give anything of value to any employee committee for the purpose of causing such a committee to influence others in their right to organize or not to organize.

Mr. Sheridan went on to give some specific exceptions to the above where: (1) the payment made to a union is in the form of a duly authorized dues check-off; (2) the payment was to a jointly administered company-union trust fund and, most important, (3) where the payment was to any employee whose regular duties include acting openly for the employer in labor and personnel matters.

On the subject of picketing, Mr. Sheridan explained that extortionate picketing is illegal and punishable by a $10,000 fine and up to 20 years in prison. Recognition or blackmail picketing is also illegal. No recognition picketing may take place where (1) the employees are already in a union and are living out the term of a contract or (2) within a period of 12 months after the N.L.R.B. has held an election in which no union has been recognized. If neigher (1) nor (2) exist, the union may picket for a period of up to 30 days. At the end of 30 days, recognition picketing must halt. Mr. Sheridan drew the conclusion that it will probably continue under these circumstances since all the union need do is show that it is "advising" the public that the employer is not organized. The union is thus engaged in "informational" or "educational" picketing.

The balance of the talk was concerned with employer reporting requirements under the new act, chief of which is the provision which requires management to report to the Department of Labor any agreement with any independent contractor where the contractor undertakes to persuade employees in their right to organize or not to organize. Any payment pursuant to such an agreement must also be reported. The major exclusion here would seem to be that the employer is not required to report the services of such a contractor by reason of his giving or agreeing to give advice to the employer.

The balance of Mr. Sheridan's speech emphasized that the new law has placed more responsibility than ever before on the shoulders of front line supervision for interpretation to employees of management's aims, goals, and desires.

W. L. Kite, *Secretary*

Ohio Section

One hundred and twenty Ohio TAPPI men attended the Oct. 15, 1959, dinner meeting at the Manchester Hotel, Middletown, Ohio.

Speaker for the evening was Samuel Alfend, chief of the Cincinnati District of the Food and Drug Administration. Mr. Alfend's talk covered the general purposes of the Food Additives Amendment of 1958 and the way the law was intended to cover indirect as well as direct additives to food. He described the type of information required in a petition seeking clearance for a given additive and the factors governing the Administration's evaluation of a petition. The wide interest in Mr. Alfend's subject was evidenced by an extended discussion period following his talk.

Program committeeman for this meeting was Gino Biasi of the National Diamond Corp.

November Meeting

One hundred and seventy-two men attended the joint dinner meeting of the Ohio Section of TAPPI and Miami Valley PIMA, held at the Manchester Hotel, Middletown, Ohio, Nov. 10, 1959.

The speaker for the evening, Edward C. Pandorf, senior engineer for the Cincinnati Gas and Electric Co., spoke on "Applications of Atomic Energy," a subject of great importance to the modern world.

Mr. Pandorf reviewed the history of discoveries in the field of radioactivity and atomic energy and described atomic structure and the mechanics of energy released by nuclear fission and fusion. He surveyed present-day industrial applications of atomic energy to the fields of medicine, biology, and agriculture with emphasis placed on increased usage in the future. Mr. Pandorf discussed the use of the heat of nuclear fission as a substitute for the carbonaceous fuels in present-day electric power production. Interesting slides illustrating modern-day atomic-electric power stations and their component equipment were shown.

The keen interest in Mr. Pandorf's subject was evidenced by an extended discussion period following his talk.

Program committeeman for the meeting was Gordon Adams of the Diamond National Corp.

George E. Martin, *Recording Secretary*

Empire State (Northern District)

October

The fifth annual mill meeting of the Northern District took place on October 8 when over 100 members toured the J. P. Lewis Co. mill and the facilities of the Fiber Products Research Center, Inc., at Beaver Falls, N. Y.

Dinner was served at Johnnie's Inn, Deferiet, N. Y.

Left to right: John L. Hudson, Jr., vice-chairman, Northern District; Rudi Kutter, Beloit Iron Works; Derry Akeroyd, Beloit Iron Works; Russell C. Clark, chairman, Northern District; Michael Papesh, Beloit Iron Works

Virginia-Carolina Section

November

The following remarks were delivered by Henry Chesley, president, Wilson Paper Box Co., Richmond, Va., at the September 18 meeting of the Virginia-Caroline Section.

When I first began to think seriously about what I might say on this occasion I jotted down a few opening remarks to the effect that it seemed strange that a manufacturer of paper cartons should be undertaking to address a group of technical experts in the paper and pulp industry, and, yet, material consideration of this situation developed the reasoning that the only possible excuse for the existence of a group such as yours is to improve product quality and to guarantee the continuance of such quality to those of us in the converting industry and to the ultimate consumers of paper products.

I not only have a great interest in this subject from the standpoint of an executive in a converting industry, but, in addition, as the largest single stockholder in my company, I have a financial interest that is put in jeopardy at times by poor quality of paper and board.

It so happens that on the very day I first began to think about what I would say to you, I was confronted with two situations that brought this subject home to me in a very forceful manner.

One of the instances involved a situation where we had sold a customer of long standing on the idea of changing the grade of paper that we were using as a wrap on his boxes. We had advocated this change because we felt we could make a better product and turn out a box that would have better appearance in self-service stores. We purchased 50,000 lb. of white offset paper from a mill owned by one of the largest paper manufacturers. This paper came to us in approximately 125 rolls and was sheeted and sent to a lithographer for printing four colors. When the job got under way, the lithographer called us and advised that he was running into terrible trouble with the paper. We sent one of our people to look at the paper and found that scattered through the run were sheets containing bad surface imperfections, fine creases, and bits of foreign material. When we contacted the mill that furnished this material, they immediately started an investigation and the next morning told us that they had discovered that rolls nos. 8 through 23 probably had these imperfections in them. I would venture to say that the man who was responsible for allowing this paper to go out of their mill is a member of your organization or he is under the supervision of such a person.

On the very same day, we received a shipment of 52 tons of folding box board from another mill. This board was supposed to be identical to 19 previous shipments that we had received from this mill for the same grade in the same size and for the same customer. The board was carefully packed on skids, wrapped with waterproofing paper and, apparently, had been handled with loving care. When we put this lot of board into production, we found, to our amazement, that over half of it had been run 0.038 thick instead of 0.043 as called for. Unfortunately for us, we did not discover this lightweight board until we were well into the run, with the result that the job had to be taken off the presses and the remaining stock carefully checked for thickness. After it was sorted, we started up again to keep our customer in production but had to stop once more until the mill could furnish more board of the correct thickness. Then, for the third time, the job had to be put on the presses and the run completed.

I cite these two instances for the simple reason that I believe that sometimes we become so thoroughly involved with our scientific and technical affairs that we forget just why we were hired in the first place. I would suspect that none of us would hold our jobs had not the people who employed us felt that by so doing we would make money for the company—certainly not cost them money.

The two instances that I refer to cost my company over $2800 and in the case of the paper supplier I have a sneaking suspicion that their loss ran in the neighborhood of $3000. In the case of the board manufacturer, he involved us in a loss that ran well over $1000 and certainly the mill must have suffered a larger loss, not to speak of the damage to goodwill in both instances.

It is a rather paradoxical situation that at this time when we are confronted with the largest demand for paper and board and products made therefrom, that we are also confronted with probably the most highly competitive market conditions that any of us have ever operated under. Our customers are demanding almost the impossible, particularly where high-speed automatic packaging machinery is involved. I can assure you with some degree of authority that unless our quality, both of raw materials and workmanship, can keep pace that we are in for serious difficulties.

The industry which I represent here tonight is confronted on all sides with competition from plastics, foils, and many other competing products. Our only salvation lies in the strict standardization of our products and a rigid adherence to quality standards. The converter is completely helpless unless his board and paper sources maintain these standards. Many of you, like myself, have been connected with the pulp and paper industry for a good many years. The progress we have seen from the old days when anything got by to the present day when quality standards demand the finest product, is almost phenomenal. With our present equipment it is possible to maintain quality in every step of our operation providing we can build quality into our personnel, both supervisory and productive. Those of us who do not maintain this quality are going to find that we will soon be out of business.

I predict that in the next two decades we will probably see developed and manufactured products that we never dreamed of before. Whether they are made of paper or whether they will be made of competing materials lies almost solely in the hands of people like yourselves.

I, for one, am a great believer in people and I honestly believe that with the proper treatment of plant personnel that we can accomplish almost the impossible. The size of today's investment in plant and equipment demands that each of us, individually, must keep pace with the technological improvements that we create, for certainly the mere creation of robotlike machines can never replace the intelligence that should be available in the men we employ to build and operate these machines. Without the men, the machines are merely worthless hulks of iron and steel. As a converter, I am more and more aware of the stark necessity for the training and development of technically minded people in our industry. I trust that you who represent this technical group realize that you have an obligation not only to the management of your plants but to the investors in them. That is why you were hired in the first place and you should never lose sight of it. If America is to prosper and to maintain its capitalistic form of democracy, it will endure just so long as the general public can get what it demands—a quality product at a lower cost.

Again, I would like to remind you that the product that we as converters turn out can never be any better than the raw materials that we work with regardless of the amount of effort we put forth. I, for one, am grateful for the technical advances that have been made and I can only plead with you to continue to make them, but most important of all, once an accomplishment is yours, never give up the effort to maintain it. These are the things that have caused the pulp and paper industry to become one of the fastest growing groups in the world, and I predict with your help we will continue to be just that.

Pacific Section

The Pacific Section held its second meeting of the year at West Linn, Ore., November 17 and had as its theme "Coating and Printing." The technical part of the program held at the West Linn High School Auditorium began at 2:00 p.m. and lasted till approximately 5:00 p.m.

Gary Jones, Weyerhaeuser Co., Longview, moderated a very fine program and one with which he personally has much knowledge and intimate contact. Five papers were presented:

1. "The Major Commercial Printing Processes," by Wm. F. Cyrus, Crown Zellerbach Corp., West Linn, Ore.
2. "Printing Inks," by Arlon Reese, Interchemical Corp., Portland, Ore.
3. "A Review of Coating Methods," by Dwayne Clark, Simpson Paper Co., Everett, Wash.
4. "Coating and Printing of Paper," by Charles Galusha, Crown Zellerbach Corp., West Linn, Ore.
5. "Coating and Printing of Paperboard," by Richard Cordingly, Weyerhaeuser Co., Longview, Wash.

The social hour followed the meeting at 5:30 p.m. at the

Members at the Pacific Section Meeting (l. to r.): Gary Jones, moderator, Weyerhaeuser Co., Longview, Wash.; Arlon Reese, Interchemical Corp., Portland, Ore.; Dwayne Clark, Simpson Paper Co., Everett, Wash.; Charles Galusha, Crown Zellerbach Corp., West Linn, Ore.; Richard Cordingly, Weyerhaeuser Co., Longview, Wash. Not shown is Wm. F. Cyrus, Crown Zellerbach Corp., West Linn, Ore., who was the first speaker

Crown Zellerbach Hotel. A specially prepared buffet dinner at 6:30 p.m. was highlighted by our guest speaker, Milton Bell of Abbott, Kerns & Bell Co., who spoke on the "Printing Industry." He pointed out the problems connected with the user of various grades and qualities of paper and gave many examples of how his industry helped sell more paper and sell better grades of paper.

Approximately 140 people participated in this meeting and Bob Gaiser, Crown Zellerbach, West Linn, did a very fine job making all the necessary arrangements.

ROBERT SMYTHE, *Secretary-Tresaurer*

Printing Process—Abstract

W. F. Cyrus

THE three principal printing processes, letterpress, offset lithography, and rotogravure, are described to provide background for the Pacific Section TAPPI meeting on "Coating and Printing." Through the use of slides, the processes are described in sufficient detail to indicate the fundamental differences between them, the means of producing tones, and an explanation of process color reproduction.

W. F. CYRUS, Crown Zellerbach Corp., West Linn Div., West Linn, Ore.

Printing Inks

Arlon Reese

PRINTING inks can trace their origin back to at least 2600 B.C. where the Chinese and Egyptians used lampblack or soot mixed with animal glue or vegetable oil to formulate their printing inks. Ink making became a highly developed art under the Chinese who were printing from hand carved blocks in the 11th century—This being 400 years before Gutenberg introduced movable type in Europe.

In the early days, every printer made his own ink. The basic ingredients were boiled linseed oil, lampblack, and some earth colors. The process was a slow one as well as a well guarded one. Every printer had his own secret formula that he would use, one that he thought was always a little bit better than the other printers in his area. Actually inkmaking was established on a commercial scale in Europe in the 16th and 17th centuries. In American colonial days, inks were imported from Europe. However some craftsmen like Benjamin Franklin still preferred to make their own. The first American ink factory was established in 1742. I feel we can skim through historical data and bring ourselves up to a reasonable time, remembering such significant discoveries as lithography in 1796 and rotogravure in the mid-19th century.

Inkmaking has seen its greatest growth and development in the last 20 or 30 years. By 1920 the age old "art" of inkmaking gave way to scientific methods. Many of us in the industry feel that the last 10 years has seen the jet age of printing inkmaking. Some of the more notable developments have been: steam or moisture-set inks, heat-set inks, quick-set inks, gloss inks, and flexographic inks. Flexographic inks could command an entire area in itself in a discussion of our modern day packaging field.

Basically what are we talking about when we speak of printing inks? They can be broken down into three parts: pigments (solids), vehicles (fluids), and driers and compounds (miscellaneous other materials).

We can remember, for the sake of continuity, that all pigments will be the same in many different classes of inks. The coloring matter does not necessarily change until you start getting into specifications calling for lightfastness, grease resistance, alkali resistance, and so forth.

The varnishes and driers, along with other additives, are what make up the different kinds of ink. For instance, you can use the same pigment in a news ink, a flexo ink, and a litho ink, yet the vehicles employed make the finished product as different as night is to day.

Pigments are the solid coloring matter in the inks, and partially determine whether the ink can be used for offset and other specific purposes such as meat wrap, butter wrap, soap labels, or, if a label, can be spirit varnished. The proper choice of pigments will allow us to make an ink that is lightfast, water bleed resistant or alcohol resistant.

Some basic dyes are also used in ink formulas setting up still another set of specifications for us to consider.

To wet the pigments we must have a vehicle or varnish. Not too many years ago we were only concerned with linseed varnishes, however today there are scores of different varnishes all with their own characteristics and uses, also many synthetics are employed here.

To our pigments and varnishes we need a drier and other compounds, waxes, plasticizers, plus other component parts to formulate a balanced ink. By now you can see that inkmaking has grown to a science that is a little beyond the realm of the average printer or pressman.

Seven major graphic arts processes are in daily use. They are: letterpress, offset, gravure, flexography, screen process, die stamping, mimeograph, and stencil.

Each of these processes requires inks that are especially adapted for the specific method at hand. In most cases the inks are not interchangeable. For example, letterpress inks cannot be used for screen process or flexo work and vice versa. Why can't we use the same ink. Let us take a closer look at a few of them and see. News ink has a consistency of a heavy oil, without any tack or drag. A screen process ink is more like a soft butter. Gravure ink has a solvent type body. In fact, gravure inks are generally reduced appreciably at the press with solvent before running. Litho reminds you of molasses in winter. Not quite as sticky, however it has a firm, heavy body. Yet each one of these inks has its own place and would not work in another process.

A news ink runs fine on news stock where it can be readily absorbed. Yet it would print like an "oily blob" on litho stock. A label ink would pick and tear newsprint to shreds.

ARLON REESE, Interchemical Corp., Portland, Ore.

This points out a very definite need for our various kinds of paper. Flexographic inks also have a fluid body and dry mainly by solvent release, such as rotogravure. The solvents employed in these determine the rate of drying. News inks on the other hand, dry by penetration only. Letterpress inks for the most part, dry by oxidation. When you consider that moisture set inks dry by precipitation you can readily understand why inks must be formulated for a specific use to run on a specified kind of paper.

Each classification can be broken down into many different types of inks. Let us take letterpress for example, we find news ink, magazine and publications, book printing, commercial printing, folding box, corrugated, bag printing, and a long list of specialty printing processes. When you include heat set inks, moisture set, hi-gloss, nonskid, spot carbon, magnetic, and several other types of formulations, you begin to see how complex an industry inkmaking has become.

Time will not permit us to even attempt to delve into the technical side of all these classifications so permit me to set up an example of how an ink is actually established for a particular job. Let us say, Smith and Company, a large carton manufacturer, calls for a special match for a new account. The ink company representative is shown the color to be matched. This can be anything from a standard color chart to a printed carton or even a piece of cloth that might have caught the fancy of a customer. The paper stock is discussed and a sample obtained. (Need Ink in a RUSH!) Layouts and specifications must be thoroughly understood by both parties. What is to be the end use of the carton? Does it have to be light-fast, grease resistant? Is it a gloss ink or is it to be overprinted? Will it run wet or dry trap? All of this verbal exchange must take place before the inkmaker can even start to get his hands dirty. As soon as everything is understood the information is passed on to the color matcher. Checking the specifications he immediately eliminates the materials not suited for the job and concerns himself only with certain pigments that fall in the proper category for the job required. Starting from a scratch mix that includes a dab of each color to get his match, he will further eliminate pigments until he arrives at an o.k. Now a formula is started and all ingredients to be used in this ink are weighed on a laboratory balance. When the color matcher reaches a point where he feels the formula is close enough to check, he pulls a proof on the specified stock and checks it against the copy. Further adjustments are made if necessary and a final proof is pulled and set out to dry. The color is again checked and if satisfactory a production formula is written and sent to the weigh-up room. The same formula is used that was developed in the laboratory only the amounts of ingredients are increased. King-sized mix masters are used to mix pigment, vehicle, solvent, driers and compounds into a homogeneous mass ready for milling. Most ink companies use three roller, water cooled, stainless steel roll mills for their grinding operation. After grinding the color is checked against the lab standard and if o.k.'d is ready for still further tests. Tack readings are taken on an inkometer, a grind gage is used to determine if all particles are ground to a fineness to enable any screen work and fine lines to print clean. Drying tests as well as rub tests are made on this production batch and proofed on the submitted stock.

You would think by now the customer could have his ink. However, in some cases, even further tests are made including spectrophotometric curves of the color to insure an accurate record of this color for all time. Now the ink is ready for the production of the cartons.

To climax our example we should be able to say the ink ran like a dream, production was at a maximum, and the customer was very pleased, but—let's be realistic and confess that we do have servicemen who are working full time taking care of jobs that run like nightmares rather than dreams. Circumstances come up that put gremlins around the press. The weather is against you and the pressman had burnt toast for breakfast. It is the serviceman's job to be able to diagnose, on the spot, the immediate troubles and make whatever adjustments are necessary to get the job back into maximum production. This example, naturally is a hypothetical one, however, one that is faced many times a week by all inkmakers. Solving printing problems is a task that combines the best efforts of press manufacturers and papermakers as well as the ink companies. Through technical associations, such as your own as well as other, F.T.A., G.T.A., L.T.F., these problems are generally brought to a successful conclusion.

Scientific research and development is constantly going on to build a better product. Many different means are employed to look into the mysteries of printing inks, presses, and paper. Some of the instruments used are the electron microscope which magnifies pigment particles until they look like rocks, color measuring devices, rub testers, and even extremely high-speed photography. We have a short film that will demonstrate how this high-speed photography is helping us solve some of our problems on ink mixting, as well as some of our customer problems. It is through cooperation and interchange of ideas and developments that better things will be brought to us in the future in order to do a better job.

Remembering that ink contains a pigment, varnish, and a drier, plus a constant look at the end use and specifications, we might conclude with a rough definition. "The lore of Benjamin Franklin combined with the art and technique of the craftsman, plus the scientific skill from the laboratory, make today's printing inks."

Presented at the meeting of the Pacific Section of TAPPI held in West Linn, Ore., Nov. 17, 1959.

A Review of Coating Methods

D. J. Clark

Although the coating of paper is not new to our industry, the major advance has been made in the last 25 years. The first attempts at coating were on wall paper, which dates back to 1850. In 1854 there was some brush coating of box lining and attempts were made to coat book paper. The coating of paper on the paper machine dates back to 1880 when attempts were made to coat at the size press. Many patents have been applied for in relation to size press coating. These are based on the use of low solids coatings and apply only light coat weights, differing from modern machine coating which employs high solids coating, and applies coat weights nearly as great as is done on conversion coaters.

For many years the brush coater was the only conversion coater, and this process is still in use in many mills. It is a very slow coating process and has many disadvantages which will be discussed later. It has been said that brush coating of paper is much more of an art than a science.

The growth in production of machine coated paper in the last 25 years is phenomenal. According to the United States Office of Commerce statistics, the production of machine coated paper in 1935 was less than 1000 tons annually. In 1945 there were 333,000 tons produced, in 1950 1,020,000 tons, and in 1955 production had jumped to 1,290,000 tons. The major factor responsible for this growth in production of coated paper was the increased use of starch as the adhesive. This was made possible through the introduction and use of modified starches. Other factors were the development of soybean proteins as coating adhesives, and the use of domestic clays and precipitated calcium carbonates as pigments.

D. J. Clark, Simpson Paper Co., Everett, Wash.

With the advent of these materials it was possible to develop coating colors which possessed the flow properties necessary for high speed production and which gave coated papers the ink absorbency, smoothness, pick strength resistance, wet rub resistance, and other characteristics required of the various printing processes.

It is the intent in this paper to discuss some of the major coating processes, stressing the inherent advantages and disadvantages relating to each, and to show schematic slides which will illustrate the mechanics of the various coating methods. My personal experiences are limited to size press and conversion air-knife coating. These methods I will cover more in detail and describe some of our problems and experiences at Simpson Paper Co.

As mentioned earlier, brush coating was the first conversion coating process, and its use is limited strictly to conversion coating. In the usual method, coating is transferred from a pick-up roll to the paper through the medium of a revolving brush. In some installations the transfer is made with a felt-covered roll rather than a revolving brush. A small driven doctor roll then removes the excess coating and does the initial smoothing. The paper then comes in contact with three series of reciprocating brushes, which spread the coating evenly over the surface of the paper. The first set of brushes are the coarsest, have the greatest amplitude, the lowest frequency, and are made of Russian bristle. The middle set of brushes act as blenders and are made from black China bristles. The last set are finishing brushes made from badger hair. After the coating has been smoothed out the paper usually is festooned and passed through a drying room. If the paper is coated both sides, it is air-floated for a distance before festooning.

Brush coating generally produces a high quality coated paper which can be supered to a good finish. There are, however, several serious disadvantages. First, the speed of coating is limited to about 400 f.p.m. because of the reciprocating brushes. Then, too, the coating tends to dry in the brushes forming lumps which drop onto the paper. The brushes have to be cleaned frequently, at least every 4 hr., and cleaning is not easy. Brush marks are common on the coated paper, but proper synchronization of the brushes will minimize this characteristic. There is also quite a serious problem with foam due to the action of the brushes. With all these disadvantages, however, there is high quality brush coated paper produced.

The next few coaters I will discuss are classified as print roll coaters. They are so named because the principles of ink distribution equipment on printing presses are employed in the distribution, smoothing, and working of the coating prior to application to the paper.

One of the best known machine coating methods is the Consolidated coater, which was developed jointly by Peter Massey and the Consolidated Water Power & Paper Co. The process was patented in 1933. This system consists of two units, one for each side of the paper. In each of these two systems are gate rolls where the coating is impounded. The coating is fed to the nip through an ocillating shower. In some installations there is an overflow from this nip and, in others, the supply to the nip is equivalent to the consumption. The coating passes through the nip of the gate rolls, and is continually split and worked from one oscillating distributor roll to the next. The last distributor roll transfers the coating to the 48-in. hollow steel rubber-covered coating rolls. The paper passes through the nip of these rolls two where the coating is transferred to the paper.

There are very important advantages to this method of coating application. It is capable of high speed production ranging from 1500 to 2000 f.p.m. The coating color is used at about 65% solids, so there is a minimum of water to dry. There are, however, several disadvantages. The physical characteristics of the raw stock are very critical, it must be slack sized to permit rapid absorption of the water, and it must be of very uniform moisture and finish smoothness to obtain a quality coated paper. This is high-speed production equipment, and is not suitable for manufacturing of small sized orders or for frequent changes of grades. The rubber rolls are easily damaged, which makes the paper machine susceptible to loss of production due to down time. Probably the greatest factor is the high initial cost of installation. It is apparent that the problems of the coating on this type of equipment are controllable, as there are thousands of tons of publication paper produced on the Consolidated coater for the periodicals which we receive in our homes weekly and monthly.

Another variation of the print roll coater is the Faeber. This method employs a large number of distributing rolls which gives excellent distribution of the coating. It is capable of high-speed production—up to 1200 f.p.m. The drying is accomplished with a single large heated drying cylinder. If the paper is coated both sides, two of the coaters are installed in series. Usually coat weights of 3 to 5 lb. per ream are applied; the principal disadvantage is the over-complexity of the system.

Another type of coating adapted from a printing process is the offset gravure. The gravure roll is etched with the desired pattern and then chrome plated. The coating is transferred from the furnish roll to the etched roll and the excess doctored off, leaving only the coating in the recessed cells in the etched roll. The suction produced by contact of the rubber roll with the etched roll transfers the coating to the rubber roll, which in turn transfers it to the paper. An important advantage of this method of coating is that there is no variation in coat weight, providing the pattern has been properly selected, and the coating formulation is properly designed for release from the cells. It has the disadvantage of lacking versatility in changing coat weights. If a change is desired, the machine must be shut down and another etched roll installed, which will give the desired coat weight. This coating process is ideal for high-speed coating day after day on the same item. When this equipment is used on the paper machine, it is installed near the dry end of the machine as solids up to 65% can be used, minimizing the drying problem. Coat weights up to 15 lb. per ream are possible.

Next is a Champion machine coater. This system employs gate rolls which are nonmetering. An excess of coating is transferred from the gate roll to the applicator roll, and the excess is doctored off by the small reverse running metering roll. It is believed that this system should take coatings up to 65% solids, and apply coat weights up to 20 lb. per ream per side. It is reported that this method of coating gives a minimum of patterning; and it would appear that the chief problem would be in the construction and operation of the small metering roll to give the proper control of the coating transfer to the applicator roll.

The Champion coater is used principally on board machines. The metering, smoothing, and doctoring of coating is accomplished by the quarter-inch wire wound rod which returns the excess to the tub. This type of coater is in wide use due to its simplicity of operation, its low initial cost, and because it occupies very little room on the paper machine. In many installations multiple units are installed, either to give multiple coatings to one side, or for the coating of both sides. Coat weights of 2 to 4 lb. per ream per unit are usually applied and the coating color is generally 40 to 45% solids.

Another method of coating, which should be mentioned, is cast coating. In this process the wet coated paper is pressed against a highly polished chrome plated drying cylinder. A very smooth and glossy surface is accomplished without supercalendering of the paper. The slow speed operation of this coating method has placed the paper in a price range higher than can be tolerated for normal printing jobs. This

paper is used primarily for speciality jobs, and represents the highest gloss and most uniform ink receptivity of any coated paper produced today.

The air knife coating process is generally used in conversion coating, although it finds application in the machine coating of board. In this process an excess of coating is transferred from the pick-up roll to the paper. The excess is doctored off and the coating smoothed out by a blast of air from the air-knife. A suction apron, or a suction roll, located at the entrance of the air tunnel maintains tension on the paper through the application and doctoring of the coating. The paper is then carried through a drying tunnel where steam heated hot air evaporates the water. The coat weight is easily controlled through the air pressure on the air doctor and by altering the speed of the coater.

There are several advantages to this type of coating application. There is absolutely no coating pattern, and a very uniform layer of coating is applied to the surface of the paper. The hills and valleys of the surface are covered more uniformly than in other methods of application; consequently, the raw stock surface does not have to be as smooth as on roll coaters. Maximum coat weights can be applied ranging up to 17 lb. per ream. The speed of the machine is limited principally by the drying capacity. In our operation we normally run at about 800 f.p.m., but speeds up to 1500 f.p.m. are possible. A considerable portion of our tonnage is on coated one-side label, which is to be varnished ultimately.

The heavier coat weights obtainable with this process makes possible supering to a high gloss and smoothness, resulting in excellent varnishability. Also, with the heavier coat weight, brightness can be improved through changes in coating formulation. Then too, it is possible to use internally sized raw stock, important to coated one side label papers, which may be soaked prior to application or may be applied to wet bottles. One of the most important advantages is that this coating method is adaptable to the manufacturing of small orders, and to rapid changes of grade. With the equipment we have, it is possible to make changes in coating color with a very minimum of lost machine time.

One serious disadvantage is that the coating color usually contains about 60% water, resulting in a high cost of drying. There is also a tendency toward skip-coating, although in our operation this has not been a serious problem. The flow characteristics of the color and rate of drying must be controlled carefully to prevent pitting of the surface. Another drawback is that only one side can be coated at a time, and the paper must be put through twice for coated-two-side.

We have had a few serious problems in air knife coating. We did have some trouble with foam in the coating and have had difficulty in finding the defoamers which would take care of the foam, without causing oil spots or pinholes in the coated surface. It took us some time to arrive at the optimum qualities of the raw stock with respect to furnish, internal sizing, surface smoothness, density, etc., and to develop the coating color formulation which gives coated one-side label paper the desired printability and satisfactory varnish hold-out.

We also do some size press coating on equipment which has been installed on two of our paper machines. It differs from a conventional size press in that a five-roll calender stack is located just ahead of the press, and a pick-up roll is used to transfer coating to the bottom roll. Also included is a combination hot air and calrod heater between the size press and the first after-drier. Most of our production is in coated book paper for letterpress printing using starch coatings. We have been able to apply 12 to 14 lb. total coating to two sides. Many months of work and experimentation was required before we were consistently manufacturing satisfactory paper. The variables which we had to investigate in the process included furnish changes, internal sizing, density, smoothness before coating, per cent coating solids, viscosity of coating, hardness and crown of size press rolls, temperature of size press rolls, and control of draw between size press and after-drier. We now have the combination of conditions and specifications which are satisfactory for our operation.

One of the newest coating processes in the industry is the trailing blade. This process is being used on both the paper machines and as a conversion coater. The normal coat weight applied by this method is 4 to 7 lb. per ream, but recent developments have indicated that normal coating weights of 14 to 15 lb. per ream are possible. There are two versions of this equipment, but the one employing a dam of coating behind the blade is the most common. In the other method the paper is immersed in the coating and the excess doctored off. Control of coat weight is accomplished through control of solids and flow characteristics of the coating.

The advantages of this process are quite numerous and significant. A very high quality coated sheet with a flat surface is obtainable, and the raw stock does not have to be as smooth as for roll coating, although it must be slack sized. This equipment can be incorporated on a paper machine at minimum expense, and can be used in conjunction with a roll coater to obtain the high quality of the double coated paper. Also, there is no practical limitation as to speed. One common problem with this method of coating application is scratches on the surface due to lumps collecting behind the blade. It is also necessary to change blades quite frequently, although this can be accomplished in a few minutes. Normally the water content of the coating is 60 to 65%, resulting in a high drying cost. With the application of light coat weights there is a minimum of coating left on the hills of the surface, so that poor varnishability and variation in ink receptivity is experienced. It is quite apparent that the advantages for this method of coating far outweigh the disadvantages, as many such installations are in use.

In closing, I should like to emphasize that the description of coating equipment is covered in numerous publications including TAPPI Monograph No. 8. Also, that we would be pleased to show any one interested, the equipment we have at Simpson Paper Co.

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The Coating and Printing of Paper

C. Galusha

Basically, paper is coated to obtain a higher surface smoothness, and thus improved print quality. Coating raw materials, coating formulation, and coating methods have all been directed over the years toward obtaining this high level of smoothness. A coated surface also displays a higher brightness, opacity, gloss, a more uniform appearance, and ink receptivity, all of which are factors included under the heading of print quality. These are the factors the printer is referring to when he describes a particular signature as having good reproduction, ink hold-out, contrast, realism, sharpness, and snap.

The methods of coating most commonly employed to obtain this higher surface smoothness on publication grades include the various roll coaters, the air knife and, most recently, blade-type coaters. The desirable feature of all three

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methods is the wide range of coat weights which can be satisfactorily applied. They differ primarily in method of controlling coat weight, in flow properties of coatings used, and in surface characteristics of the actual coating film.

Certainly, the primary control of coat weight for all three methods is through coating solids. The finer control is accomplished through speed of the gate rolls for roll-type coaters, through air pressure for air knife coaters, and through head and blade adjustment for blade-type coaters.

Coating rheology for the three methods differs quite markedly. While air knife coatings are free flowing, low viscosity colors, roll-type coaters perform most satisfactorily with relatively viscous colors exhibiting a high degree of thixotropy. Colors for blade units lie between these two extremes depending on solids, speed, and coat weight applied.

Both roll and air knife coaters tend to follow the roughness of the base sheet. Roll coaters have a further disadvantage in that a characteristic pattern results from the film splitting at the coating nip. Blade-type coaters have a surface filling action which is responsible for the levelness and smoothness obtained. It is not unreasonable to expect a smoothness gain two to three times over that of any other coating method. This one factor is responsible for the great concern and popularity which has been directed toward blade coaters the last several years.

Publication grades include papers for sheet-fed offset, wet offset, letterpress, and rotogravure. Each printing process requires a particular set of paper characteristics which are necessary to obtain good printability and high print quality. The papermaker realizes these characteristics stem from both the base sheet and coating applied, and is very much aware of the difficulties the printer is sure to encounter when they are absent. He maintains these characteristics through fiber furnish and formation, proper moisture control, proper coating formulation, and the use of special dust prevention machinery.

It might be well at this time to mention that in many cases the paper is not the only source for the defects the printer encounters. Such defects may also stem from improper selection of ink, in regard to tack and drying for the paper being used, and also from faulty press adjustments. In many cases a thorough analysis of the problem is required to definitely establish the source of trouble.

Following is a discussion of the more important paper characteristics necessary to obtain good printability and print quality under the various printing processes along with exhibits to illustrate several of the defects commonly encountered.

SHEET FED OFFSET

Papers for sheet fed offset must be strong to withstand the high tack inks and gripper action of the press; flat to avoid misregister and wrinkling; free of lint and dust to avoid hickies, spots, and piling tendencies; and uniformly but not overreceptive to ink to avoid mottle and ink drying problems.

The furnish for offset papers generally include a high percentage of long-fibered chemical pulps to maintain the desired level of strength and minimize fiber pick. The coating for offset papers requires some degree of insolubilization since moisture is transferred from the blanket to the coated surface. This insolubilization is accomplished through the selection of adhesive materials which naturally render insoluble coatings, or which can be treated and made to render such coatings. The need for insolubilization is particularly true when multicolor printing is involved since the nonprinting areas on one unit become printing areas on succeeding units.

To minimize dusting problems, offset coatings generally contain relatively high levels of adhesives which have high pigment binding qualities. Such coatings often contain lubricants and plasticizers to further reduce dusting tendencies and improve flexibility of the coated surface. In addition such equipment as infrared heaters, silicone and teflon-covered driers, as well as sweat driers are employed to prevent coating from adhering to the drying equipment and later being picked up by the damp coated surface. In some cases the dry coated web is passed through a set of vacuum brushes to remove all traces of loose surface dust as further assurance toward the production of a dust free sheet.

WEB FED OFFSET

Paper characteristics for web offset are comparable to those required for sheet fed offset. However, there is sufficient evidence that coatings for web fed papers need not be insolubilized because of the higher speed and lower level of fountain solution required for the particular type of inks used. These factors plus the advantage of printing both sides simultaneously places this method of offset printing in a very attractive position.

The inks used for web printing are generally heat setting and are less viscous and tacky than for regular offset.

Due to the intense heat the sheet encounters in passing through the drying chamber after printing, strength and moisture control are important to provide sufficient resistance against splitting at the fold and blistering. Blistering occurs from the sudden expansion of air and moisture under a sealed surface, resulting from both the coating and ink film, when the printed sheet is subjected to the unusually high drying temperature. Web papers should be well formed and contain a low level of moisture to prevent fiber clots and moisture pockets. Also, coat weights should be relatively low to minimize the degree of surface sealing.

LETTERPRESS

Since letterpress papers are printed in the absence of a rubber cushion, these grades require the maximum degree of softness and smoothness. Such quality is obtained principally through the base sheet by proper fiber selection and formation. Smoothness is further developed by including various pigment fillers. Ash content of the base sheet generally averages 5 to 6%.

Coat weights for letterpress grades generally range between 6 to 10 lb. per side depending on the over-all weight. Starch adhesives are most commonly used since there is no requirement for insolubilization. Starch-latex adhesives are also used for special finishing properties.

Defects resulting from loose surface dust are generally of a lesser problem for letterpress printing than for offset. Surface dust is generally passed on to the inking system, but hickie problems may be encountered if such material should later settle on the plate. Also, large amounts of fine coating dust absorbed into the inking system can cause a "graying out" condition, or a reduction in color intensity.

Both smoothness and ink hold-out are important factors to provide cleanliness and clarity, and to avoid breaking and "run together" in the halftone areas. Ink hold-out is desirable for good print quality, since color intensity of letterpress inks are generally lower, and over-absorbency will cause the colors to gray out. The greater part of publication printing is done by letterpress.

ROTOGRAVURE

Rotogravure papers should have a tight formation obtained through proper selection of short and medium length fibers. Like letterpress, the highest quality is obtained through surface smoothness and softness of the over-all sheet.

Ash content will generally range between 12 and 20% for development of maximum smoothness. With this base sheet smoothness, relatively low coat weights are required (generally ranging between 3 to 5 lb. per side) and the re-

sulting coated surface provides an absorptive bed for the thin solvent type inks used, and all the characteristics the printer includes under the heading of print quality.

Hickies and Spots

Hickies may be caused by ink scum, coating dust, or foreign material attached to the blanket or plate. Particles of these various materials are ink receptive, but hold the blanket away from the plate near the point of contact, thus printing as a solid center surrounded with a halo.

Spots may be caused by slitter dust, coating pick with fibers attached, or other water receptive particles. Such material in contact with the plate or blanket may print as hickies at first, but after becoming saturated with water will repel ink and print as open areas or spots. Coating pick is best determined by thumbing through successive signatures prior to and after the defect was encountered to establish whether or not picking occurred. In many cases, both hickie and spot defects require a microscopic examination of the particles removed from the plate or blanket to properly identify the material.

Piling

A piling problem generally occurring on coated papers having an insufficient degree of water insolubility. The coating gradually builds up on the blanket and causes "snowflaky" solids in the printing areas, and may desensitize the plate in the nonprinting areas.

A second characteristic required for sheet fed papers is proper moisture content. Moisture content of the paper at time of manufacture is generally 5 to 6%. Higher levels of moisture causes blackening when the sheet is supercalendered. A proper moisture balance between the paper and pressroom atmosphere is all important to prevent the occurrence of wavy and tight edge paper. The paper must reach the pressroom with the proper moisture level and free of any tight or wavy edge condition. If the sheets are not perfectly flat at the time of printing, misregister and wrinkling will usually be encountered. Temperature and relative humidity of paper and pressroom should always be checked by the printer before the paper is unwrapped to make certain the proper balance exists.

Misregister

Misregister problems often result from an improper balance of paper moisture with pressroom humidity. The signature in four-color printed on a two-color press shows the yellow and blue, first and second colors down, outside of the red and black. This can also be seen from the register marks at the corner of the sheet. Between the first and second runs on the press, the paper developed a slight condition of edge waviness. Under the uniform compression of the backing cylinder, the tail of the sheet fanned out and printed out of register as described. A severe wavy edge condition will cause wrinkles to form near the back and run all the way to the back edge.

Tight Edge Wrinkles

Another defect results from a tight edge baggy center condition. Under uniform compression the tail of the sheet is fanned in and causes wrinkles to form from the center toward the back. Wrinkles from tight edges, however, do not reach the back edge.

Ink drying problems are in many cases confined to the ink. However, paper moisture and pH also have an effect. Ink drying is retarded by high moisture and low pH, and papers for this process should be near or on the alkaline side. Sheet fed papers should also be well formed and provide uniform ink absorbency to avoid mottle and offsetting problems. The paper must absorb enough of the vehicle from the ink film to properly set the ink and prevent the pigment from adhering to the back of the following sheet. Other problems in drying are related to the ink and can be controlled by the printer through the use of metallic driers.

Blistering

Even though the sheet may contain the ideal properties to minimize a blistering defect, the difficulty may still occur because of the necessity of extreme drying temperature in relation to speed and ink coverage. Certainly, any high quality paper can be made to blister if suddenly subjected to an unreasonably high temperature.

Breaking and Run Together

Breaking and filling in the halftone areas occur from nonuniform surface smoothness. When the printer increases his ink level of compression to remedy the breaking in the rougher areas, filling between the dots often occurs in the higher areas of the sheet.

Roughness and Ink Absorbency

A rough surface condition caused by the coating piling due to an overdried sheet. This defect is often difficult to see on the unprinted sheet, but shows up very clearly after printing because of the difference in ink absorbency over the rough coated surface.

In conclusion, we are all aware of the unusually high print quality obtained through the process of double coating on off-machine operations. We are also aware of the pronounced improvement which can be obtained through the use of blade coating units. Because of their surface filling advantage over other methods of coating, blade units will undoubtedly be employed for the greater part of the publication paper production. This does not mean, however, that present coating equipment will be replaced. In many cases such equipment may be used in conjunction with the blades as precoating or surface-sizing equipment on one or both sides of the sheet. The ultimate in printability and development of print quality, however, will be obtained with minimum coat weight levels through the use of double blade units for both sides of a surface-sized sheet. These various methods of double coating for on-machine operations should give a degree of quality never before obtained on publication papers, and will undoubtedly be accepted by the printer with open arms.

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Coating of Bleached Paperboard for Improved Printability

Richard H. Cordingly

WITHIN the past few years, there has been a tremendous growth of coated bleached paperboard, and the future promises still further expansion of this and related industries. The primary impetus behind this expansion is the beneficial effects coatings have on printability of the treated sheet as compared with a noncoated board. The coating generally provides a smoother and more uniformly ink receptive surface than can be otherwise obtained. As a result, converters of the board are often able to produce printed products such as frozen food containers with superior appearance at a lower cost than can be obtained with other competitive materials. Other objectives for coating bleached paperboard which can be mentioned but which lie outside the scope of this paper are (1) for decorative purposes, (2) for functional purposes, and (3) for utilization of lower grades of furnish than would otherwise be required, or conversely up-grading products made from lower grades of stock thereby tending to improve the profit situation.

The large growth rate of coated paperboard could not have

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occurred without the development of modern, efficient on-machine coaters. These coaters were first developed during the 1930's by producers of printing grades of paper. Producers of board were able to borrow these methods and have subsequently adapted them to suit their own particular circumstances.

On-machine coated paperboard first became significant in 1954. Initially, roll coaters, both letterpress and offset in principle, and air knife coaters were used. Approximately one year later, the trailing blade coater began to assume a significant position in the industry. Since that time, the roll types of coaters have dropped in importance while the use of the latter two types has increased very rapidly. At the present time, the interest of the industry appears to be centered primarily on the trailing blade type.

The United States of America recently experienced a recession; and during this period of lowered industrial activity, the coated bleached paperboard industry noted an excess capacity as did many other industries. The nation's economy has recovered and the demand for coated paperboard has increased accordingly. However, the capacity still far exceeds the demand. The future, therefore, is somewhat hard to foresee over the short period, but in the long run, it would appear that the demand will steadily grow. The future depends to a great extent, however, on the wisdom of the industry in how it meets competition. It is the job of the entire industry to think, work, and discover in order to assure continued growth and expanding markets.

As politicians have learned to know history in order to meet rationally many of the national and international problems which exist, the coated paperboard manufacturer must know the history of his industry so that he also may rationally treat his problems. A very brief introduction to the coated paperboard industry has been presented earlier in this paper and the recent article by William H. Aiken is recommended for further details. The experiences of the Weyerhaeuser Co. in the Longview, Wash., mill are typical of the industry with regard to the development of commercial scale on-machine coating of bleached paperboard. In the fall of 1954, an offset gravure type of coater was installed on the bleached paperboard machine of the Longview mill. The primary advantages of the roll type of coater are: (1) coatings of high solids content can be used and relatively heavy coating weights can be applied and (2) mechanical operating problems are fairly minor. The most serious disadvantage is concerned with the problem commonly referred to as turkey track or pattern.

The potential effects of pattern on printing results are well known. Even when the pattern has been reduced in magnitude so that it is almost invisible to the naked eye, it has a very deleterious effect on the printability. One of the primary purposes in obtaining an offset type of roll coater was in the hope that pattern would be minimized.

Coating pattern on board presents a very different problem than that found on light-weight papers. In the latter case, the pattern problem can be alleviated by supercalendering, but supercalendering of board is not economically feasible and has a deleterious effect on other qualities of the sheet. Thus, the problem of minimizing coating pattern on board had to be approached in other ways.

The personnel of the Weyerhaeuser organization have thoroughly studied the theoretical and practical aspects of minimizing coating pattern and its effects on coated board printability. While many of the findings with roll coater applications are specific in nature, others are of general application to all types of coating methods. In brief, their studies showed that two factors are of primary importance in the development of suitable coated board: namely, (1) coating weight and (2) coating rheological properties. Other factors which are of secondary or unknown importance are (3) speed of coating application, (4) cohesive strength of the coating, (5) surface properties of the base stock, (6) angle of departure from the coating nip, (7) pressure of coating application, (8) diameter of the applying roll, and (9) hardness of the applying roll.

Considering the two primary factors, the influence of coating weight was found to be the greatest of all factors in affecting pattern. Pattern size diminishes with decreasing coating weight. There is, however, a practical limit below which coating weight cannot be reduced. Decreasing coating weight naturally tends to reduce the other beneficial effects to be derived from the coating process. Thus, after the minimum coating weight has been established, the other main avenue of approach assumes primary significance.

At this point, a very brief description of the types of flow which are commonly encountered is warranted. The rheological properties of fluid materials are conveniently described by plotting the relationship between the rate of shear and shearing force. Curves depicting this relationship are known as rheograms. In the case of some fluids such as water and the lower weight hydrocarbons, the rheograms consist of straight lines passing through the origin. The rheograms of many other materials do not exhibit straight line relationships and, accordingly, the viscosity of the fluid is dependent upon the rate of shear to which the material is subjected. If the rheogram is concave upward, the flow characteristic is called pseudoplastic and the viscosity decreases as the rate of shear increases. The converse type of flow characteristic is called dilatancy, and the viscosity of materials possessing this characteristic increases with increasing rates of shear.

Two other characteristics often encountered in rheological studies which are worthy of mention are concerned with (1) the yield value and (2) thixotropy. In many instances, a certain finite shear force must be applied before any rate of shear can be detected. This minimum force which must be applied is called the yield value, and probably is related to a type of gel structure which must be broken down before the system will flow.

When collecting the data for rheological studies, it is common practice to obtain information as the shearing force is both increased and then decreased. In many instances, such a procedure will result in the rheogram exhibiting a hysteresis loop, and materials exhibiting this type of flow characteristic are called thixotropic. Some workers in the field of paper coating believe that some degree of thixotropy is required if a coating is to have good leveling properties.

Extensive laboratory and field investigations showed that for the Weyerhaeuser Co.'s operation, the rheological properties of coatings should be such that at low rates of shear, the viscosity was low, and, conversely, at high rates of shear, the viscosity was high. The former condition is required in order to perform the necessary mechanical functions of pumping, screening, etc. Such a flow characteristic is also desirable from the standpoint that some flow might occur immediately after the application process and thereby lessen the effects of pattern. Conversely, a high coating viscosity at the rates of shear which are believed to exist in the coater nip will cause the coating film to split into a very fine pattern. These characteristics are most closely fulfilled by a coating having Newtonian or slightly dilatant characteristics.

Considering the question of rate of shear at the coating nip, a question which is of importance not only in roll coating but also in air knife and trailing blade methods, there appears to be very little concrete information available; however, the shear rates are probably very high. Thus, a problem in all coating evaluations is that concerned with measuring the rheological properties of coatings at extremely high rates of shear. One of the best methods presently available involves the use of the Hercules Hi Shear viscometer. This instrument, which attains very high rates of shear, is provided with a means by which a continuous curve relating shear stress

and shearing force can be obtained. It was found, however, that two coatings having essentially the same rheograms and fluid retention characteristics possessed very different patterning characteristics. Because of such observations, it is the belief of the Weyerhaeuser Co.'s research staff that the instrument may not attain rates of shear comparable to those which exist in coating operations.

The Brookfield viscometer is quite useful in coating evaluation even though its rate of shear is very low. Its utility, therefore, does not lie in the field of predicting how the coating will behave at the nip, but in indicating the ease of handling the coating prior to application.

Through extensive investigations, the Weyerhaeuser Co. personnel were able to develop coatings and methods of coating application which lowered the degree of pattern so that it was almost invisible to the human eye. Such coated boards still exhibit poor printability, however, and it was decided that methods other than roll coatings should be investigated.

The Weyerhaeuser Co. has never had an air-knife coater on the machine in the Longview mill. The company has had, however, an opportunity to evaluate this coating process by means of the facilities in the White Pigeon, Mich., operation.

The principle of air-knife coating is relatively simple; namely, an excess of coating is applied to the sheet and the excess is blown off with a stream of air. The advantages to this process are (1) the coatings tend to be applied fairly uniformly in thickness with the attendant advantage of uniform ink receptivity; (2) the coatings may be used over a wide range of solids content; (3) the coating weights can be high, an advantage which is very important in functional types of coatings; and (4) the coater is simple in design and operation. Probably its greatest advantage is its high degree of versatility. Its primary disadvantages are (1) its tendency to cause streaking if the air outlets become partially blocked, (2) its tendency to form pattern though of a much smaller degree than roll coaters, and (3) it is not a good leveler. With regard to the final disadvantage, the coating tends to follow the contours of the base sheet. For this reason among others, it was decided to use a trailing blade type of coater which has outstanding leveling characteristics. In conclusion, the air knife coating process is a very good method, and for certain applications such as functional coatings, it is superior to the trailing blade. However, the Longview personnel believed that it was not the best method for the development of surface smoothness, and therefore, printability.

Thus, in the fall of 1957, after the roll coating method had proved inadequate and the inquiries which indicated that the air-knife process was not the best method to develop surface smoothness, a trailing blade coater was installed on the bleached paperboard machine in the Longview mill. The results of the installation have been very gratifying, and demonstrate the fundamental soundness of the process since the unit was designed and built by Weyerhaeuser personnel whose primary purpose is the manufacture of pulp and paper products rather than of coating equipment.

The principle of trailing blade coating is simple and similar to air-knife coating; i.e., an excess of coating is applied and doctored off the sheet with a blade. Its primary advantages include (1) its excellent leveling action, (2) its efficient use of coating, and (3) its ability to prepare sheets with superior printing smoothness. The primary disadvantages to the trailing blade coating process include (1) its low coating weight which limits its utility in functional coating, (2) its tendency to cause streaks, and (3) its tendency to cause mottle unless the coating and base sheet are very nearly the same in color and brightness.

One of the primary factors in trailing blade coating is recognition that to produce a superior coated paperboard, the base sheet must be of superior quality. This factor is of greater importance with the trailing blade than with the roll or air-knife coaters because of the lower coating weights generally employed with the former. Thus, such factors as formation and smoothness of the base sheet must be carefully controlled for optimum results. One factor which has never been completely resolved and which certainly is open for debate, is that of which side of the sheet should be coated. At Longview, the wire side is coated for two primary reasons. Preliminary investigations indicated that the wire side of the sheet as made at Longview was slightly more receptive. The other and undoubtedly more important factor was that the installation design was more simple than that which would be required to coat the felt side.

In order to assure optimum results, treatment of the base sheet prior to the coating must be balanced for the coating in use and the final use of the coated paperboard. While in essence, such treatment starts at the wet-end of the machine and before, the size press and calender treatments prior to coating are of great importance. These treatments must be designed to maintain or preferably to improve the receptivity of the base sheet. Simultaneously, they should be designed to minimize surface fuss which could result in coating streaks. Developmental work in this field is continuous and it is obvious that no one set of treatments can be considered as the ultimate. More work, both of fundamental and applied nature, is needed in this area.

The influence of the type and nature of drier felts required in successful trailing blade coating is fairly well understood throughout the industry. The felt must be designed to minimize felt hair on the base sheet which can cause serious streaking difficulties.

Coating formulations are complex in detail and often very dependent upon a given mill's circumstances and certainly upon the consumer's requirements. In general, a typical formulation would contain about 85% pigment and 15% adhesive. The pigments consist of high grade clays, titanium dioxide, calcium carbonate, etc. The adhesives in use consist not only of natural products such as starch, casein, alpha-protein, etc., but also various synthetic polymers. The latter types of adhesives are becoming more important as they constitute a wider variety from which to choose in order to develop any given desired property. In addition, they help to improve bending characteristics, ink receptivity, and so forth. In general, the coatings are formulated to possess Newtonian or slightly pseudoplastic flow properties.

In order to control streaking, the Longview mill has found that the coating solids should be about 62% or below. As was the case discussed with regard to roll coatings, there is a practical lower limit to the coating weight which can be applied.

Because of the low coating weights which are applied with the trailing blade, it is necessary that the color and brightness of the coating and base sheet be approximately the same. If the difference is too large, serious mottle will result. In addition, excessive dry calendering of the coated board aggravates the mottle problem.

The testing of coated paperboard is, of course, a problem of extreme importance. In order to determine the printing characteristics of coated board, the Longview mill relies primarily on the IGT print, pick, and, to a lesser degree, the wet pick tests. The IGT print is used as a routine control test even though the method is somewhat laborious for, at the present time, no other test appears to be simpler and yields as valuable information. The standard IGT equipment has been amply described in many papers, and the Longview apparatus has been modified in but one respect. A motor drive was installed on the print tester so that the revolving sector could be driven at a constant speed. The print test is repeated until incomplete coverage is noted on the test samples. The ink film thickness, which has been carefully

calculated from the number of strips tested, the area of the printing rolls, and the area of the inking rolls, is recorded as the print test result. A good correlation has been found between the test results so determined and actual field test results.

The dry pick test is on less firm grounds than is the print test, and is, therefore, used to a lesser extent in coated board evaluation. The standard IGT test apparatus is used together with IPI tack rated inks. In this procedure, the sample is tested using no. 6 ink if possible as the correlation between the results obtained with the various inks has not been fully established. The sector of the IGT apparatus is motivated in this test by the spring mechanism provided on the machine. The speed of the sector at various points along its path of travel has been carefully determined so that when the test sample is inspected, the speed at the point of coating break-up can be easily determined by measuring the distance from the origin of the test to the break-up point and referring to a reference graph. The point of coating break-up has been arbitrarily designed as the first area of 1 sq. cm. in which ten picked spots can be detected. The results are recorded as a combination of two numbers; namely, the grade of ink and the speed at which break-up occurs.

Correlation of the pick test results and actual field results presents a difficult problem since each printing plant operates differently, to a greater or lesser degree, than any other plant. Even within a given plant, there may be a large degree of variation in printing presses, speeds, inks, etc. In general, however, pick does not appear to be too great a problem when using the gravure method of printing, but for letterpress and offset printing, the pick values should be about 6 to 100 and 6 to 150, respectively.

The wet pick, as originally devised by the Dow Chemical Co. and modified by the Weyerhaeuser Co., is used primarily to determine the degree of picking or milking of pigment from the sheet which will occur during offset printing. The test is conducted in a manner similar to the print test except that the sheet is dampened with water just prior to coming into contact with the printing roll. The test sample is compared to an arbitrarily chosen set of standards and given a number corresponding to the standard which it most closely resembles. The correlation between the wet pick test results and actual field results has not been fully established at the present time.

The methods used in printing coated paperboard are essentially the same as used on printing grades of paper; that is, letterpress, offset, and gravure methods are commonly employed. In the letterpress process, the printing smoothness as determined by the IGT test is of very great importance. The sheet must come into contact with the raised portions of the printing plate or ink transfer will not be complete. If the printing smoothness is not adequate, the press man can either increase the pressure or thickness of the ink film. The pressure can be increased only so far, however, since punching may result. Similarly, increasing the ink film thickness reduces ink mileage and tends to aggravate offset problems.

In the offset printing process, the ink is taken from the printing plate to the sheet by means of a rubber blanket. As a result, printing smoothness is not as important a factor as in letterpress printing. The board must have good ink receptivity, however, in order to prevent the ink from building up on the plates and blanket. As indicated earlier, board must have greater surface strength for offset printing than for letterpress printing since in the former case inks of higher viscosity are used. Coated paperboard for offset printing must also be able to resist picking and milking in the moistened state since the transfer blanket is slightly wet from fountain water in the nonprinting areas. If the coating is not moisture resistant, the loosened coating may pile on the blanket and may eventually work back into the ink supply causing considerable difficulties.

The gravure type of printing process utilizes inks of very low viscosity so that the surface strength of coated board to be printed is not of extreme importance. The question of surface smoothness is more debatable as some printers require board with smoothness superior to letterpress while others do not.

Presented at the meeting of the Pacific Coast Section of TAPPI held in West Linn, Ore., Nov. 17, 1959.

Lake States Section

The following papers were presented at the Lake States Section C. J. West Award competition, and received Honorable Mention. The competition was held May 12, 1959, in Menasha, Wis.

Our Experience with a Spent Sulfite Liquor Lagooning Operation

J. Marshall Buehler

In order to continue operation of its 130-ton sulfite pulp mill at Port Edwards and yet reduce its pollutional load to the Wisconsin River, the Nekoosa-Edwards Paper Co. inaugurated a spent sulfite liquor lagooning program in 1952.

Port Edwards is located on the Wisconsin River along a section of the stream heavily loaded with industrial and municipal wastes. Therefore, the Wisconsin Committee on Water Pollution issued orders to the industry in this area, in an attempt to improve the quality of the stream. Accordingly, in the summer of 1952, Nekoosa-Edwards Paper Co.'s proposal to construct a seepage lagoon was approved by the State Committee on Water Pollution. An experimental test hole, about 8 ft. square and 4 ft. deep, was excavated on a 70 acre island in the river just east of the Port Edwards mill. This test hole was located in a bark-filled area and spent sulfite liquor was dumped into it from a tank car loaded at the mill. The rate of seepage into the ground was determined and from these data it was determined that two ponds 40 by 50 ft. and about 4 ft. deep, would be needed to take care of the volume of liquor to be ponded. Just to take care of any excess, a 40 by 100-ft. overflow pond was also excavated. The two smaller ponds would discharge into this larger pond through a shallow overflow ditch rather than overflow their banks.

While these ponds were being opened, Technical Department personnel established stream sampling stations about 1 mile above the Port Edwards mill at a dam, and another three miles below the mill at a bridge. Other stations were also established but their purpose was only to augment data collected at the two aforementioned stations. Periodic samples were collected at these points over a 3-month period. From the water samples collected, dissolved oxygen, biochemical oxygen demand, spent sulfite liquor solids, pH, and temperature were obtained. Standard methods of analysis were used for all these determinations. The data thus collected during this three month preponding period were to be used for calculating the increase in biochemical oxygen demand loading as the stream passed the Port Edwards mill. This figure would then be used as a base figure for comparing future biochemical oxygen demand increments in this same area, thereby evaluating the effects of the ponding program.

METHOD OF OPERATION

Spent sulfite liquor is drained from the blowpits through a common header pipe to a 300-g.p.m. pump which pumps it to a 36,000-gal. steel storage tank. The liquor is pumped for a period of 1 hr. following a digester blow on each of three

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small, 10-ton capacity digesters and 1½ hrs. on a larger, 15-ton capacity digester. From the steel storage tank, the liquor is pumped at a rate of about 175 g.p.m. through 3700 ft. of stainless steel pipe to the lagoons. Part of the 4-in. pipeline is buried and part of it lies above ground. A piece of 4-in. rubber hose attached to the end of the pipe permitted use of either of the two ponds. Operation of the pumps was made the responsibility of the blowpit man. All valves are of a rubber-lined type or stainless steel, and pumps are constructed of bronze. An air connection was installed on each pump so that compressed air could be blown through the system to prevent freeze-ups during winter shutdowns.

PONDING OBSERVATIONS

About 10 days after the start-up in the fall of 1952, it was noted that an old drainage pipe on the island, neglected for years, was increasing its volume of flow and the effluent as increasing in spent sulfite liquor solids, indicating that our liquor was taking a short cut to the river rather than spreading out in a uniform seepage pattern. Therefore, an effort was made to stop the flow in this pipe. Cinders were dumped into an open crib at the upper end of the pipe. When this failed, we went to the outlet of the pipe and poured a block of concrete in front of it. Then a manhole where this particular pipe crossed our Nepco Lake water supply pipe began to overflow. So, we dug down to the drainage pipe and cut into it. Next we built a retaining dike around this hole. Thus a new pond was created to collect seepage liquor. About this time, we realized that our 36-in. cast iron pipeline, supplying the Port Edwards mill with process water from Nepco Lake, was lying in an acidic liquor bath. Fearing detrimental results, it was decided to abandon the area and seek a new ponding location, further north on the same island.

Accordingly, test holes were dug around the island, digging down to the water table in each case. Finally, a new location was found where the water level was about 5 ft. below the surface. A new pond, about 42,000 sq. ft. in area, was excavated in the sandy soil. A tile pipeline was connected to the end of the already existing stainless steel pipelines and ponding was again resumed—for a 20 min. period. By this time our tile pipe had broken in about six places due to expansion of the pipe from the hot liquor. Stainless steel pipe was then substituted for the tile and, in August of 1953, ponding was again in full operation and has continued since then.

However, a new problem now faced us. The sandy soil of the new ponds did not drain as rapidly as the bark filled did and the pond was rapidly filling. Accordingly a second and slightly smaller pond was opened. Rapid filling of these two ponds prompted the diking of a swale of land to the north and another to the east of the ponding area, thereby creating two more ponds. All four ponds were connected to the liquor distribution system with the necessary pipes and valves. Liquor was soon detected seeping into a slough connecting with the river. Therefore, a bulldozer was employed to dike off this slough from the river, thereby creating a fifth pond. However, this fifth pond was not connected to the liquor distribution system since it was intended to collect seepage liquor from the other ponds. Total area now in ponds was and still is about 5 acres.

SPRAY OPERATIONS

In the summer of 1955, an experimental spray operation was begun. A single sprinkler was installed in a grass covered sandy area. A pump was installed to pump liquor from one of the ponds, through aluminum irrigation pipe to the sprinkler head. It was our intent to determine how much liquor could be applied to the ground before flooding occurred, how long it would take the ground to recover and be ready for further use, and the effects on vegetation. Impressed with the favorable data acquired from this single sprinkler, it was decided that sprinkling of liquor would be an added benefit to our stream pollution abatement program. Therefore, three more sprinklers were added in 1957. Spraying is now carried out during the summer months only and augments the seepage ponds during critical river conditions. During the summer of 1958 liquor was sprayed on a 2-acre plot in 74 days. This was not continuous since the system is shut down on those weekends when the mill is not operating. These 2-day shutdowns each week provided sufficient time for the ground to recover from its state of liquor saturation. Approximately 30,000 gal. of liquor were sprayed each day of operation.

COLLECTION OF DATA

In order that the amount of liquor being ponded each day could be determined, the rate of the pump removing the liquor from the blowpit was determined. A totalizing clock was installed on the electrical control of the motor operating the pump, thereby recording the number of hours of operation of this unit. From this data, the volume of liquor removed from the blowpit could be determined. While it is recognized that the rate of the pump can vary from blow to blow, periodic checks of this rate show it to maintain a fairly reproducible average rate. During the pumping period, a small continuous sample is bled off the pump and collected in a 300-gal. tank. The contents of this tank are then mixed once a day and a sample removed for spent sulfite liquor solids determination. This procedure was kept up for nearly 2 years. During this time, the concentration did not vary more than 1% one way or another and was found to be proportionately related to the volume of liquor ponded. Thus a series of concentration values based on the volume of liquor ponded was established, thereby eliminating the continuous sampling. Knowing the volume of liquor ponded, the tonnage of pulp produced and assuming a value of 1.25 tons of spent liquor solids for each ton of pulp produced, we were in a position to determine what per cent of our total spent liquor solids were ponded.

Weekly biochemical oxygen demand samples are still taken. Once each month these data are tabulated and compared to the standard preponding period. An improvement in stream conditions is reported if the pollutional load of the river is not increased more than it was in the preponding period as the stream passes the Port Edwards mill.

RESULTS OF THE PROGRAM

After 6 years of ponding, we have noted the following effects. Biochemical oxygen demand data indicate that the increase in B.O.D. loading from the station above our mill to the station below the mill has varied from month to month. On some occasions a considerable improvement has been noted, while other months there is not so notable an improvement. There have even been a few months when the biochemical oxygen demand load was greater than it was during the preponding base period. However, over-all experience during the 6-year period shows a 43% reduction in biochemical oxygen demand pick-up when compared to the preponding period. This has been accomplished by the ponding of an average of about 6 million gals. of spent sulfite liquor each month, or about 46% of our total available spent liquor solids. Fiber loss through the blowpit sewer has been reduced 49%, this fiber now being deposited in the lagoons.

All vegetation in the area has been killed. Seepage rates have increased, demonstrating the dispersing action of the liquor. No odor problems have been encountered, although there is a combination sulfur dioxide and fermentation odor in the immediate area. A monthly report covering all the ponding operations, stream data, and liquor data is forwarded to the Wisconsin Committee on Water Pollution. It should be pointed out that the ponds are not dumped during periods of high stream flow. In other words, our ponds are of the seepage type and are not holding ponds.

CONCLUSION

The ponding of spent sulfite liquor which was formerly

discharged to the stream, has effected an improvement to the stream when compared to a standard preponding period. This is due to the combination of factors: (1) Sugars in the spent liquor are acted upon by bacteria present in the soil, thereby reducing the pollutional load of the liquor. (2) The liquor by being sprayed in the air or held in a pond is given a chance to get rid of free sulfur dioxide, another contributing component to the pollutional load. (3) Fiber loss to the stream is reduced by the filtering action of the soil. (4) Liquor is added to the stream in a continuous, gradual manner rather than by intermittent slugging of the stream as was formerly the practice. (5) The liquor is added to the stream, in our particular case, at a point where the river bed is rocky, turbulent, and shallow, thereby providing additional aeration of water and liquor and causing more rapid stream recovery.

Other processes for the treatment of the spent sulfite liquor have constantly been under study, and we will continue to look for a better method of treating the spent liquor. However, until a feasible method is developed, the lagooning method of treatment will definitely have its place in the improvement of our stream.

A. C. J. West Award paper presented at the meeting of the Lake States Section of TAPPI held in Menasha, Wis., on May 12, 1959.

Production Hydrosulfite Bleaching of Aspen Groundwood

Edward Masak, Jr.

THE use of aspen (*Populus tremuloides* and *P. grandidentata*) in the mechanically pulping process has enabled the paper industry to utilize a wood species that comprises one-half of the standing pulp wood in the State of Wisconsin.* Thus, aspen groundwood becomes very attractive from a conservation and from a transportation cost standpoint for the Late State paper industry. Understandably, the use of a new species brings about problems in storing, grinding, cleaning, bleaching, and proper paper machine utilization. Since 1956, the Kimberly mill of Kimberly-Clark has pioneered much of the production scale hydrosulfite bleaching of aspen groundwood. This paper will briefly summarize this work. As is the case with much technical work, time does not permit a complete research approach. Many times it was deemed sufficient to prove a theory without going into the academic approach of why the theory was true.

A brief description of the Kimberly mill groundwood system is as follows: Aspen is drum barked, cut into lengths, and ground on Sandy-Hill pocket grinders. After cleaning the initial or unbleached brightness is measured.† The groundwood is either bleached with single-stage calcium hypochlorite (which will not be discussed here) or with one of the hydrosulfites. At present, sodium hydrosulfite is used. The unbleached groundwood and hydrosulfite solution are mixed and pumped through an up-flow tower. Retention time is 3 to 6 hr. After bleaching the groundwood is washed with fresh water and delivered to the paper mill in slush form. Bleached brightnesses are determined at the paper mill.

THE USE OF ZINC HYDROSULFITE

Bleaching Response

At the Kimberly mill, zinc hydrosulfite was the first reducing type bleaching agent used. Table I indicates the weekly average bleaching response for a period of Oct. 8, 1956, to July 20, 1957. Bleaching temperatures were 45 to 55°C.

Table I. Bleaching of Fresh Poplar to Zinc Hydrosulfite Bleaching

Brightness gain required	*Zinc hydro. per ton of pulp, lb.*	*Zinc hydro. per ton per point gained, lb.*
4.0	15	3.75
3.5	$9^1/_2$	2.75
3.0	$5^1/_4$	1.75

Over this period, the average brightness gain was 3.16 G.E. points. The average consumption of 9.95 lb. per ton ($2.18/ton) was equivalent to 3.15 lb. per ton/per point of brightness gain.

Effect of Yard Stored Aspen Upon Zinc Hydrosulfite

At the Kimberly mill, peeled or rough aspen can either be used directly as fresh incoming wood or stored in the woodyard. Since groundwood made from longer stored aspen caused color, foam, and white water settling problems in the paper mill system, a study was made of the effect of this older wood on bleaching. Table II indicates as the per cent of 2-yr. old stored peeled aspen increased, the zinc hydrosulfite consumption also greatly increased. Thus, zinc hydrosulfite had very little effect as a bleaching agent for this older wood.

Table II. Effect of 2-Yr. Old Peeled Aspen on Zinc Hydrosulfite Consumption in the Bleachery

Two-year stored aspen, %	*Zinc/ton, lb.*	*Zinc/ton/point G.E. gained, lb.*
10	7.0	1.8
15	10.0	2.4
20	12.5	3.2
25	15.0	4.0
30	18.0	4.5
35	20.0	5.5

Nine month average for all aspen was 9.95 lb. zinc per ton or 3.15 lb. per zinc per ton per point G.E. gained.

To verify these production findings, the following laboratory experiment was performed: groundwood made from either fresh aspen, 1-yr. old aspen or 2-yr. old yard stored aspen was procured. Several aliquots of each groundwood equivalent to 20 air dry g. of each pulp were weighed out, placed in plastic bags, evacuated with carbon dioxide gas, mixed with appropriate amounts of freshly made 1% zinc hydrosulfite solution (0.94% by titration with indigotin disulfonic acid), re-evacuated with CO_2. The bags were then sealed and placed in a 50 ± 0.5°C. constant temperature both for 2 hr. Brightnesses were determined on the bleached pulp.

Table III indicates that 2-yr. old stored wood reached a maximum bleached brightness using 14 lb. of zinc hydrosulfite per air dry ton of groundwood. The bleach response of 2-yr. old stored wood at this 14-lb. level of 2.4 points is equivalent to the bleach response of fresh aspen at 6 lb. per ton.

Fresh aspen and year old aspen have the same pounds per ton per point G.E. gained bleaching response as has been shown to be the case with spruce and balsam groundwood.* Thus for production purposes, the 2-yr. old stored aspen was "unbleachable" and had to be consumed in the sulfite process.

Zinc Hydrosulfite Bleaching of Defective Aspen

In order to determine the effect of various Aspen defects, species, and age upon bleaching, $2^1/_2$ to $3^1/_2$ cord samples of freshly cut aspen containing various defects were ground on a production grinder. A sample of freshly cut Balm of Gilead and a sample of aspen that had been stored for 9 months in the woodyard were also ground. Samples of the groundwood were bleached under CO_2 for $2^1/_2$ hr. at 50°C.

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* *Wisconsin Forest Facts*, 1959 Edition, Wisconsin Forest Industries Committee, Green Bay, Wis.

† All brightnesses data, unless otherwise noted, were made on pulps that have not been modified in pH. That is, the pulp "as is" is found in production. Handsheets are made according to TAPPI.

* Whitman, Frank A., *Tappi* 40 No. 1: 20–24 (1957).

Table III. The Effect of Aspen Age Upon Laboratory Bleachings

Zinc hydro./ton, lb.	Fresh aspen			1-yr. peeled			2-yr. peeled		
	Brightness	Δ	lb./t/pt.	Brightness	Δ	lb./t/pt.	Brightness	Δ	lb./t/pt.
0	67.6	..	..	62.5	..	..	60.4	..	..
6	70.0	2.4	2.5	..	..	..	61.6	1.2	5.0
10	71.1	3.5	2.86	66.1	3.6	2.8	62.3	1.9	5.3
14	72.0	4.4	3.2	..	..	..	62.8	2.4	5.8
15	..	..	..	67.4	4.9	3.18	..	..	..
22	..	..	..	68.2	5.7	3.5	62.7	2.3	8.7

using 22 lb. of zinc hydrosulfite per ton of air-dry pulp. An excess of bleaching agent was used to insure that any difference in bleaching was due to the defect and not to experimental error.

Table IV indicates that rots of both the white and red variety decrease bleaching response. Surface defects and dirt due to excessive knots are also very difficult to bleach. The rot defects, surface defects, and excessive knots produced a low unbleached brightness wood. The combination of low brightness and poor bleaching response would make it advisable to place restrictions on levels of such defects on incoming aspen.

Neither stained aspen nor Balm of Gilead affected the bleaching response adversely. Here again, the unbleached brightness was very low because of this and minimum amounts of such defects or species were to be tolerated.

Yard storing aspen over a summer caused the unbleached brightness to decrease 6.8 G.E. brightness points. The bleaching response was equivalent to freshly cut poplar. Economics dictated that freshly cut aspen of higher unbleached brightness should be used whenever possible.

THE USE OF SODIUM HYDROSULFITE

Bleaching Response

A production trial was performed in which sodium hydrosulfite was used in place of zinc hydrosulfite. Table V indicates the response of aspen to sodium hydrosulfite was much better than to zinc hydrosulfite. A decision was made to use sodium hydrosulfite in production. Table VI bore out the trial conclusions.

The cost of bleaching with sodium hydrosulfite was one-half that of bleaching with zinc hydrosulfite. It is not within the scope of this project to determine the reason for this phenomenon, it is sufficient to take advantage of it.

Effect of Yard Stored Aspen and Balm of Gilead Upon Sodium Hydrosulfite Consumption

In May, 1958, freshly cut samples of rough aspen, peeled aspen, rough Balm of Gilead, and peeled Balm of Gilead were cut. These were stored in the Kimberly mill woodyard. Periodically, a sample of 2 to 3 cords of each was processed through the woodroom and ground. The unbleached brightnesses of the resultant groundwood samples were determined.

Table IV. Effect of Defects Upon Bleaching with Zinc Hydrosulfite

Defect	Defect, %	Unbleached brightness	Bleached brightness	Gain
Red rot (*Radulum casearium*)	8.7	63.7	67.7	4.0
White rot (*Fomes igniarius*)	14.1	61.8	63.6	1.8
Excessive knots	3.3	62.4	66.3	3.9
Surface defects, cankers, cracks	4.4	60.7	61.0	0.3
Stained, internal	14.6	57.7	62.8	5.1
Aged, 9 months over a summer	1.1	64.7	70.2	5.5
Balm of Gilead (*Populus balsamifera*)	21.5	58.2	63.9	5.7
Control				
Mixture of big tooth (Populus tremuloides) and trembling aspen (*Populus grandidentata*)	1.2	71.5	76.5	5.0

Samples bleached with 22-ib. zinc hydrosulfite per ton for 2½ hr. at 50°C. under CO_2.

Samples of wood were obtained and aspen defects were determined by D. Prielipp and L. Moore, Kimberly-Clark of Michigan.

Table V. Bleaching Response of Fresh Aspen to Sodium Hydrosulfite

Points gain in brightness desired	Sodium hydro. per ton, lb.	Sodium/ton/point brightness, lb.
3.5	3.5	1.0
4.0	4.3	1.1
5.0	6.0	1.2
6.0	10.0	1.7

The samples were then lab bleached with 8 lb. of sodium hydrosulfite for 2 hr. at 60°C. under nitrogen.

Table VII indicates that Balm of Gilead had a bleached brightness 10 points lower than aspen. Rough aspen did not lose brightness as rapidly as did peeled aspen. Rough aspen of higher brightness could be stored for 2 months before more than 1 point was lost, while the peeled aspen lost 2 points in as little as 2 months. After 4 months storage, the brightness loss was essentially the same for all aspens and remained constant. The rough Balm had essentially the same loss as did peeled aspen. The results on peeled Balm of Gilead are not conclusive.

Table VI. A Comparison of Production Costs of Zinc and Sodium Hydrosulfite Bleaching

	Bleaching Agent	
	Zinc-hydro. Sept. 1956–July 1957	Sodium hydro. Aug. 1957–Feb. 1959
Bleaching brightness gain	3.16	3.79
Hydrosulfite per ton, lb.	9.95	4.74
Dollars/ton	$2.18	$1.09
Hydro/ton/point gained, lb.	3.15	1.24
Costs of bleaching agent: Zinc hydrosulfite, $0.2129/lb. Sodium hydrosulfite, $0.2313/lb.		

In order to verify some of these conclusion 11 cars (a 3-day supply) of freshly cut rough aspen containing 14% Balm of Gilead were processed through the Kimberly mill woodroom and pulp mill. Preceding this, 3 days of freshly cut rough aspen was used. Following the balm, two days of a mixture of year old and fresh rough aspen was used.

Table VIII indicates the added expense in bleaching Balm of Gilead, and yard-stored wood. Thus, from an economic standpoint, it is best to bleach freshly cut aspen of low balm content. Storage should be done in the rough rather than the peeled form especially if storage time is to be less than 4 months in duration.

BRIGHTNESS REVERSION OF HYDROSULFITE BLEACHED GROUNDWOOD

Reversion of brightness is common in groundwood pulps.

Table VII. The Sodium Hydro Bleaching of Aged Aspen and Balm of Gilead

(All Brightnesses Are Sulfite Sodium Bleached Brightnesses)

Storage age of wood	Rough aspen		Peeled aspen		Rough balm		Peeled balm	
	Brightness	Loss in brightness upon storage	Brightness	Loss	Brightness	Loss	Brightness	Loss
1 month	76.14	0	75.71	0	63.00	0	64.78	0
1½ months	75.26	−0.088	75.64	−0.07	61.25	−1.75	64.84	+0.06
2 months	75.39	−0.75	73.75	−1.96	60.22	−2.78	65.44	+0.66
2½ months	75.0	−1.14	73.8	−1.91	61.90	−1.10	64.40	−0.38
3¼ months	74.58	−1.56	73.80	−1.91	62.52	−0.48	64.33	−0.45
4¼ months	72.87	−3.27	72.60	3.11	61.55	1.45	64.59	0.19
5¼ months	74.53	−1.61	73.76	−1.95	62.51	−0.49	64.51	−0.27
6½ months	73.1	−3.0	74.4	−1.31	61.5	−1.50	65.1	+0.32

Aspen and Balm samples were cut in Upper Michigan in May and delivered to Kimberly in June. The first trial (1 month old storage) was made in May. D. Prielipp and L. Moore, Kimberly-Clark of Michigan, were in charge of wood procurement for this trial.
All samples were bleached with 8 lb. sodium hydro per ton under nitrogn for 2 hr. at 60% at Research and Development, Kimberly-Clark, under the direction of G. Saunders.

This reversion is especially noticeable in hydrosulfite bleached groundwood in contact with ion.* It has been hypothesized that the iron is reduced by the hydrosulfite to its colorless ferrous form. After contact with an oxidizing agent, air being the most common, the iron is plated out on the pulp in its dark brown ferric form. This cases an apparent reversion of the groundwood.

A method of testing for this reversion is through the use of an EMF (oxidation-reduction) and pH meter since reversion takes place at different EMF's at different pH's. The Kimberly mill system was checked with an EMF and pH meter. Table IX indicates that reversion takes place upon washing hydrosulfite bleached groundwood, upon the addition of alum prior to the paper machine fan pump and upon dilution at the fan pump.

Table VIII. Use of Balm of Gilead and 1-Year Old Aspen in the Sodium Hydrosulfite Bleachery

Wood	Brightness gain	Sodium/ton, lb.	Lb./ton/point gained
Fresh rough aspen	-2.5	1.4	0.47
14% balm	3.65	3.0[a]	0.82
50% year-old peeled aspen	5.05	7.7	1.53

Since the Balm was bleached 0.5 point higher in brightness than the other samples, it is estimated that 2.65 lb. of sodium per ton was used.
[a] Per cent of Balm was determined by R. Eggen, Kimberly-Clark of Michigan.

Table IX. Oxidation-Reduction Data of a Mill Groundwood Stock System

Location	Time	pH	EMF, (millivolts)	EMF range at which iron reversion takes place at this pH
Unbleached	8:00	7.0	+140	−400 to +500
Tower	9:10	6.2	−490	−300 to +500
Washed	9:15	7.1	+70	−400 to +500
Virgin fiber mixture	9:30	7.1	+50	−400 to +500
V.F. + broke	9:30	6.9	+40	−400 to +500
V.F. + broke + alum	9:30	4.3	+160	+100 to +500
V.F. + broke + alum + dilution for headbox	9:30	4.2	+230	+150 to +500
Headbox	9:45	5.1	+200	−40 to +500
Wire trim	9:45	4.9	+230	0 to +500

EMF ranges of iron reversion determined by G. Saunders, Research and Development.

This indication of reversion as shown by oxidation-reduction was further verifield by actually testing brightnesses at indicated points of reversion.

* Yankowski, A. A., *Tappi* 40, No. 9: 717–20 (1957).

Reversion of Hydrosulfite Bleached Groundwood at the Washer

The reversion upon washing was tested by procuring paired samples before and after the washer. Brightnesses were determined immediately after sampling and after allowing the samples to sit with occasional stirring for 5 hr., the approximate time in the bleached hydrosulfite bleached storage tank. A 1.2 points of reversion is found on the stored washed samples while only a 0.2 point reversion took place in the unwashed samples as shown by Table X.

The brightness of the two immediate samples were the same, indicating reversion is a rather slow reaction and not instantaneous. The use of 3 to 5 lb. of EDTA per ton of groundwood prevented the reversion of washed groundwood.

Table X. Reversion of Hydrosulfite Bleached Groundwood Upon Washing and Storage

Sample	Brightness		Reversion	Dissolved solids (g./100 ml. of effluent)
	Immediate	5 hr. storage		
Unwashed	68.0	67.8	0.2	0.1850
Washed	68.0	66.8	1.2	0.0624
Washed + 3 lb. of EDTA per ton of groundwood	68.0	67.9	0.1	...
Washed + 5 lb. of EDTA per ton of groundwood	68.0	68.0	0	...

Consistency of both samples was 4.66 to 4.78.

Reversion at the Paper Machines Due to Alum

In order to verify the reversion at the paper machines as indicated by the oxidation-reduction data an experiment was performed in which the brightness of hydrosulfite bleached groundwood was determined at its normal pH of 6.8 to 7.0. Aliquots were then adjusted to 4.5 pH with sulfuric acid or papermaker's alum or iron free alum and brightnesses were determined. This was repeated with a paper machine stock.

Table XI indicates that 1-point of brightness reversion could be prevented through the use of iron free alum. A month long mill trial of iron free alum verified these results. This trial will not be discussed in this report.

Further work is progressing at present using EDTA treated alum and low iron content aluminate.

A. C. J. West Award paper presented at the Meeting of the Lake States Section of TAPPI held in Menasha, Wis., on May 12, 1959.

Work on a production problem such as this could only be possible due to the combined efforts of many men in many departments. Thanks go to A. C. McIntyre and W. McDonald, Kimberly Pulp Department; D. L. Wolfe and R. J. Greenwalt, Kimberly Technical Department; A. A. Yankowski and G. Saunders, Research and Development; R. Eggen, D. Prielipp and L. Moore, Woodlands; H. Pierce, Kimberly Mill Manager; and P. R. Eastwood, Kimberly Production Manager.

Table XI. Effect of Various pH Adjusting Agents Upon Reversion

Stock	BRIGHTNESSES							
	As is brightness (pH 6.8–7.0)	Sulfuric acid adjusted	ΔH_2SO_4 − as is	Papermaker's alum	ΔH_2SO_4 − pm alum	Iron free alum	ΔH_2SO_4 − if alum	Δ pm Alum − if alum
Sodium	68.6	71.0	+2.4	66.4	−4.6	67.4	−3.6	+1.0
Mixed stock	71.1	73.4	+2.1	69.3	−4.1	70.4	−3.0	+1.1

An Approach to Continuous Cooking

Wavell Cowan

THE application of a continuous process to the digestion of wood in the production of pulp should be a desirable improvement over the batch system. Certainly a continuous process has many inherent advantages over a batch process. The reduced size of the necessary primary equipment makes closer control of such variables as temperature, pressure, and chemical concentration possible. But, more important, it becomes economically feasible to alter these conditions at any desired time during the cooking reaction.

Existing continuous cooking processes have not proved sufficiently effective to cause obsolescence of the batch system. The attempt has been made to utilize the practical knowledge of the normal batch cooking operation together with various unique mechanical arrangements to produce a continuous system. The flexibility that might be successfully incorporated has been largely ignored. The reason for this is the lack of fundamental understanding of the chemical processes involved in pulping reactions.

The wide diversity of opinion expressed in the literature on cooking mechanisms and the effect of cooking variables, each apparently supported by experimental observation, inexorably points to the need for wholesale reassessment in this area. The realization that the problems inherent in any cooking process will not be solved by one grand, sweeping experiment but only by painstaking, long-term research is probably the first necessary forward step in this re-evaluation.

In this paper an attempt is made to develop an appreciation for the primary concepts upon which the ultimate success of continuous cooking will depend.

In developing the picture presented in the following pages, primary significance appears to be given to the alkaline cooking process. This in no way is meant to suggest that this area is potentially more important to continuous cooking than such other processes as acid sulfite or neutral sulfite pulping. Rather, this analysis should be considered as representative of the approach that must be applied to all areas until specific knowledge limits the usefulness of any particular area.

ANALYSIS

To achieve a maximum economic significance, continuous cooking must utilize significantly faster pulping rates than are normally employed in the conventional batch operation. How this can be accomplished is the first question requiring major consideration. The pulp quality as obtained by continuous cooking must at least be comparable to present pulp quality. This is a second point of major importance. The ability to control pulp quality or define pulp characteristics over a wide range is a final high desirable feature which should be considered in any approach to developing continuous cooking.

A brief examination of what is involved in a normal cooking operation is probably a good starting point for this discussion. Wood in chip form is charged into a digester followed by cooking liquor, after which the temperature is raised. The interaction between the cooking liquor and the wood chips is of primary interest to this analysis. In gross terms, the desirable effect is a dissolution of lignin from the wood through the action of the cooking liquor. The undesirable effect is the degradation and removal of carbohydrate material. These reactions require the following sequence of events:

1. The cooking liquor must first penetrate into the macrostructure of the chip.
2. Reactive molecules (or ions) in the cooking liquor must undergo microdiffusion* to a solid-liquid reaction interface.
3. An absorption of these reactive molecules on the solid surface must occur.
4. Chemical reaction between the solid phase and adsorbed reactive material can then take place.
5. The reaction products must then desorb, suffer microdiffusion from the reaction interface, and then diffuse through the macrostructure of the chip into the surrounding cooking liquor.

The rate of these individual steps will depend upon the local conditions (in a micro-sense) of temperature, of ionic and molecular species including type and concentration, of electrokinetic environment, and of the physical and chemical nature of the wood. These, then, represent the variables affecting the over-all pulping rate. However, a number of them lose significance when interest is directed toward *major* increases in pulping rate. Certainly the chemical nature of the wood is largely beyond control. Although the physical nature can be somewhat altered, these changes cannot be expected to have any major effect on pulping rate. The electrokinetic environment is established by the components of the system and so is not an independent variable. Thus, consideration need only be given to the chemical make-up of the cooking liquor and to the temperature at which the pulping reaction is carried out.

A vast number of pulping agents, both inorganic and organic have been investigated, and no evidence exists to support a view that substantially better pulping rates can be economically achieved by substituting a new pulping agent for existing ones. Similarly there is no evidence that a catalyst of significant importance will ever be developed as an answer to rapid pulping requirements.

Considerable work has been done attempting to define the effect of chemical concentration and cooking temperature on pulping rate. With few exceptions little attention has been paid to extraneous variables. As a result obvious discrepancies have occurred, and many of the conclusions drawn are of questionable value. However, certain trends have been established which give at least a qualitative picture. These can be illustrated from the work of Hobden (*1*) with alkaline (NaOH) cooking of eucalyptus. This work has particular merit as will be discussed later. An increased delignification rate is the result of increased concentration of alkali and increased pulping temperature, and a much larger rate increase can be expected by elevating the temperature than can be effected by corresponding increases in alkali concentration.

Elevated temperatures thus appear to be the answer to the required rapid pulping essential to continuous cooking. This is not an unexpected conclusion. A 10°C. rise in pulping temperature is generally believed to double the delignification rate. Larocque and Maass (*2*) in alkaline cooking experiments point out that the temperature coefficient ranges from 1.7 to 2.7 per 10°C. temperature rise at 160°C. Recent work by Walters (*3*) with neutral sulfite cooking liquor suggests that this temperature coefficient is even higher (approx. 4) at higher temperatures (210°C.). Thus it appears certain that high temperature cooking is a desirable basic factor around which to build a continuous cooking process.

This brings us to the second point, that of pulp quality. Critics will vociferously maintain that high temperature cooking precludes the possibility of producing quality pulp. Their argument is that degradation of carbohydrate material is excessive at high temperatures and dominates the improved delignification rate achieved. I do not believe that this assertion has ever been adequately verified experimentally. Furthermore, there is an indication that such a view is incorrect.

Experimental work aiming at the determination of the effect of high temperature cooking on pulp quality has suffered from two major drawbacks. The first is that care has not been taken to eliminate the penetration effect from influencing the results. Improperly penetrated chips subjected to high cooking temperatures will yield degraded pulp, but the

* This term refers to movement across a thin film adjacent to the actual solid surface.

degradation can clearly not be totally ascribed to the high temperature. Secondly, the actual cooking operation is not conducted under approximately isothermal conditions. A significant portion of the total cooking time is represented by a heating-up period. Here again results cannot be accounted for entirely by the period at which the maximum temperature is operative. In effect, only for experiments in which penetration has been carefully excluded as a limiting factor and where the cooking is essentially isothermal can legitimate conclusions concerning high temperature cooking be drawn.

Walters (*3*) in work previously mentioned, designed his experiments to fulfill the above requirements. Using isothermal cooking conditions with aspen wafers, he clearly demonstrated that a neutral sulfite cook conducted at temperatures up to 210°C. will not cause serious degradation of the pulp produced. The work of Hobden (*1*), also referred to previously, is another example where required experimental conditions were met. Although he did not conduct his cooks at temperatures above 170°C., it is of interest to consider the trends his data establish.

The results indicated are that the total carbohydrate content of the pulp is *higher* for a given lignin content when the delignification is achieved at a higher temperature. This trend cannot be extrapolated to higher temperatures with any validity, but there is certainly no indication in these data that higher temperatures would have a deleterious effect on pulp quality as judged by carbohydrate content. The only possible conclusion is that a need exists for definitive work in this area of high temperature cooking. However, the prospects for successful high temperature cooking are not as dark as many people might believe.

A problem relative to high temperature cooking which is implied in the previous section is that of initial penetration of the liquor into the wood chip. Good penetration is essential for the production of uniform quality pulp. Where cooking time is conceived in minutes instead of hours, the time involved in achieving adequate penetration assumes great significance.

Stone (*4*) distinguishes two distinct mechanisms for permeation of a liquor into the gross woody structure of the chip. *Penetration* is the advance of a liquor-air or liquor-void interface under the influence of a pressure differential. *Diffusion* signifies the advance of liquor into a water or liquor saturated structure as a result of a concentration gradient. For clarity, these distinctions are adopted for subsequent discussion.

The time necessary to ensure adequate penetration will be primarily dependent on the nature of the capillary structure of the wood chip involved, which defines the resistance, and the differential pressure causing penetration, which is the driving force. Secondary variables are the temperature and nature of the penetrating liquor. In the study by Stone (*4*) oven-dry 1-in. wood cubes were evacuated and flooded with deaerated water under vacuum. The time for sinkage to occur is suggested as a qualitative measure of penetration time. For different species of hardwood, sapwood specimens had sinkage times of $^1/_2$ to 9 min.; heartwood, of 16 min. to 8 days. The inference is that penetration time is a definite problem where rapid continuous cooking is envisaged.

Diffusion rate is a function of the concentration gradient and the total cross section available for diffusion. Temperature has a marked effect on diffusion processes and so is an important variable. The actual capillary structure is of less importance since it is the overall area contribution and not the pore distribution which is important. Lusby and Maass (*5*) in diffusion studies using sodium hydroxide and sprucewood conclude that almost instantaneous diffusion would occur under the elevated temperature conditions of a normal cooking operation. Thus, it is possible that diffusion offers a more promising prospect than does penetration for achieving rapid initial permeation of cooking liquor into the chip structure. However, diffusion presupposes a preliminary penetration stage and so the penetration problem must eventually be faced.

The idea of opening up the wood structure by some form of mechanical pretreatment offers the possibility of marked reduction of penetration time. However, lowering of pulp quality has been traced to many forms of mechanical pretreatment and definite evidence is presented (*6*) to demonstrate the marked decrease in pulp strength as a result of severe crushing of sprucewood. Evidently, further study in this area should attempt to define the reasons for such effects as a prelude to the possible development of a more satisfactory wood pretreatment.

Another alternative is to substitute the wood wafer for the wood chip. The main problem here is probably the technical development of an economical wafer producer although some concern should be given to any detrimental pulp quality that may result from this substitution.

It was suggested that control of chemical type, concentration, and pulping temperature as a function of pulping time are conceivable goals for continuous cooking. Such control, if properly defined, might produce better absolute pulp quality in addition to introducing the means of varying pulp characteristics as desired.

No one denies the complexity and multiplicity of chemical reactions involved in any cooking operation. Existing studies have to a large extent dealt with these complexities in a superficial manner. As a result of this limited research effort we are in no position to appraise the following possibilities. At several particular stages of a cooking process it might be highly advantageous to alter the cooking environment by (1) changing the cooking temperature, (2) altering the chemical concentration, or (3) introducing or substituting a second pulping agent.

These questions will never be answered by empirical experiment as there are simply too many possible combinations to be tested. What is needed is fundamental knowledge concerning all the chemical reactions involved and the rate-controlling mechanisms that describe them. Such knowledge can only be slowly evolved by a continuous, methodical, and meticulous fundamental research program.

CONCLUSION

Continuous cooking offers inherent advantages of flexibility and control with the possible resulting prospect of improved pulp quality and control of pulp characteristics. For maximum economic benefits continuous cooking must utilize rapid pulping rates. The best prospect for accomplishing this appears in the area of high temperature pulping. The accompanying problem of possible pulp degradation must be definitively explored. The factor of penetration rate which becomes limiting suggests the use of a mechanical pretreatment of wood or substitution of a wafer for a wood chip. Both such possible solutions require careful research and development. Finally, the ultimate utilization of possible flexibility and control can only result from a fundamental research program seeking knowledge of basic reaction mechanisms involved in wood pulping.

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Applications of Atomic Energy

Edward C. Pandorf

THIS talk reviews the history of discoveries in the field of radioactivity and atomic energy, and describes atomic structure and the mechanics of energy release due to nuclear fission reaction. Industrial applications of atomic energy are surveyed, and the current status of atomic, electric power productions is described and illustrated.

HISTORICAL REVIEW

The concept that matter is made up of tiny particles dates back at least 2500 years to ancient Greek and Hindu philosophers. In the 5th century B.C. Democritus named these particles "atomos," but no further progress was made in this theory until 1808 when John Dalton, an English school teacher wrote a treatise on the atom as a unit of chemical structure. In 1869 D. I. Mendelyeev published a study on the weights of elements which resulted in the Periodic Table of Elements. The discovery of x-rays by W. C. Roentgen in 1895 stimulated further investigation into the subject of radioactivity. In 1896 H. A. Becquerel discovered natural radioactivity in uranium ore. Marie and Pierre Curie named the phenomenon "radioactivity" in 1898, and isolated the elements radium and polonium. By 1904 Ernest Rutherford had identified 20 elements which possessed radioactive properties. In 1919, Rutherford demonstrated the principle of transmutation by converting nitrogen to oxygen by alpha particle bombardment. This was probably more important than the objectives of the medieval alchemists who strove to transmute lead to gold. In 1932 James Chadwick proved the existence of the neutron as an elementary particle, which solved many problems which had puzzled the scientists, and which also furnished the key to releasing energy from an atomic nucleus.

The history of atomic power starts with Albert Einstein who theorized in 1905 that energy and mass are intraconvertible in accordance with his expression "$E = mc^2$." In 1934 Enrico Fermi performed experiments involving bombardment of uranium with neutrons. He reported curious results which he could not interpret. In January, 1939, O. R. Frisch and Lisa Meitner, German refugees working in Denmark, explained the process of nuclear fission. The Frisch-Meitner theory was brought to the United States on Jan. 16, 1939 by Niels Bohr, and was confirmed within a few weeks by laboratory experiments at Columbia, Carnegie Institute, Johns Hopkins, and the University of California. Almost immediately the possibility of a nuclear chain reaction was realized with the vast potentials of either controlled or uncontrolled power release. Under pressure of wartime development the first self-sustained nuclear chain reaction was accomplished on Dec. 2, 1942 at the University of Chicago; and on July 16, 1945 the first atomic bomb was detonated at Alamogordo Flats, N. M. A large number of scientists and companies responsible for the success of the military nuclear program were anxious to be released from their wartime assignments, and were happy to turn their talents toward the peaceful aspects of atomic energy.

NUCLEAR STRUCTURE

All materials on the Earth are made up of combinations of the 92 natural elements plus tiny quantities of the dozen or so of the heavy, man-made elements. The smallest particle of a chemical compound is a molecule, which in turn is made up of atoms of the compounding elements. An atom comprises a nucleus within orbital electrons. The nucleus is built up of two major kinds of particles—protons and neutrons. The distinguishing feature of an atom of each element is the number of portons in its nucleus. This determines the atomic number of the element, and also the number of orbital electrons surrounding the nucleus of a normal atom. A proton carries a positive electric charge, and an orbital electron carries a negative electric charge. With equal numbers of each, a normal atom has no net charge.

A neutron is about the same weight as a proton, but has no electrical charge. The atomic weight of any atom is due almost entirely to the protons and neutrons in its nucleus. Different nuclei of atoms of one element may contain different numbers of neutrons, forming isotopes of the element. Isotopes of one element always have essentially indetical chemical properties, but may have different nuclear properties.

The smallest particle of substance visible through a modern optical microscope will weigh about a 100-billionth part of a gram, and will contain about a billion atoms, which seems to indicate the improbability of ever being able to see an individual atom. With atomic diameters on the order of 4 billionths of an inch, most of this space is used by the orbital electrons, making the nucleus tiny indeed. Upscaling the nucleus of a hydrogen atom to the size of a baseball would increase the radius of the electron orbit to something over three miles! Despite the unbelievably small dimensions of these atomic particles, a lot has been learned about their characteristics and peculiar properties.

FISSION ENERGY

According to the Einstein equation, 1 lb. of material is equivalent to 11.4 billion kw.-hr. of energy. The fissioning or splitting of the nucleus of an atom of uranium-235 occurs almost instantly after that nucleus absorbs a free neutron. The resulting fission products are two lighter nuclei and two or three free neutrons. The weight of all these products is about 99.9% of the weight before fission. In the process 0.1% of mass is converted into energy in the forms of fast-moving fission products and radiation. Bombardment of surrounding materials increases molecular motion, which results in increased temperature.

INDUSTRIAL APPLICATIONS

The naturally occurring isotopes of elements are mostly nonradioactive or stable varieties; although a few, like potassium 40, are unstable, and throw off radioactive emanations to achieve stability. The naturally occurring heavy elements like radium and uranium have no stable isotopes, and they very gradually decay through many unstable steps until the ends of the decay chains at stable isotopes of lead. Radioactive emanations or radiation can be in several forms involving either minute particles or rays. All of these radiations are detectable by instruments, but none of our body senses is capable of recognizing them.

All fission products are radioactive isotopes, and any element which is subjected to neutron bombardment is converted into a radioisotope by induced radioactivity. We read of the hazards of these materials, particularly those present in bomb fallout, but may not realize the many practical and valuable applications of radioactive materials.

In medicine and biology radioactive tracers are in general use for diagnosis and for helping us learn more about the functioning of living things. Also for arresting certain diseases the use of large scale radiation therapy is proving effective.

In agriculture, tracer techniques, insect and disease control, and induced mutations all use radiation sources.

Industrial applications of radiation are mainly based on these three characteristics:

1. Indications of presence of radioactive materials.
2. Penetration of radiation through solid materials.
3. Effects of radiation on other materials.

The first category includes tracer techniques: leak detection, wear, corrosion, wetting, diffusion of alloying constituents, and many others. The penetration characteristic is the

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basis of x-ray and isotope radiography for making photographs through visually opaque materials. The measurement of attenuation by materials being penetrated is the basis of many gaging functions, such as measurement of thickness, density, and level. Industrial irradiation of materials may become very important in food processing, but now finds greatest application in catalytic refinery processes and in the polymerization of plastics.

HEAT AND ELECTRIC POWER

While some of more subtle and perhaps interesting or valuable industrial uses of radioactive materials may still await discovery, the most obvious use is the substitution of the heat of nuclear fission for the carbonaceous fuels which the world is burning up at ever increasing rates. Many studies and predictions have indicated approximate dates by which economically recoverable supplies of gas, oil, and coal will be exhausted. Long before these dates are reached there will be regional shortages for which the economic solution will be the highly concentrated energy of atomic fuel.

In the United States there are two incentives for research and development in the field of atomic-electric power. The first is for manufacturers of industrial equipment to sell complete plants and hardware to customers in those parts of the world where atomic energy will soon compete economically with conventional fuels. The second incentive is economical large scale heat and power production in this country, in order to supply these services by whatever means is most attractive to the companies' customers and shareholders. While our nuclear development is still far from competitive at this time, the power industry has the responsibility for supporting reasonable experimental costs for the design, construction, and operation of nuclear plants in preparation for the time when these plants will be economically integrated into our power systems.

Presented at the meeting of the Ohio Section of TAPPI held in Middletown, Ohio, Nov. 10, 1959.

Empire State (Northern District)

Progress Report on Inverform Development

Derry Akeroyd

Beloit Iron Works has for a long time been interested in the development of what is referred to as "controlled formation." As you know, with the conventional vat, stock is furnished to a mold and formation begins to take place immediately. However, a goodly amount of study by a number of people has been done and it is still an educated guess as to most exactly what takes place as the stock continues on its route down and around the vat crucible. Forming, washing, reforming, peculiar flow patterns—all have their effects on the finished sheet. The fourdrinier sheet is also subjected to a variety of forces as it travels along the wire. After being turned loose on the traveling wire, the sheet undergoes all types of rearranging as it has to contend with forming boards, table rolls, deflectors, and suction boxes. Two-sidedness and wire mark are just a couple of the more well known results of the rearranging that can be done to a sheet before its mat is fixed.

The subject of "controlled formation" or the process of setting the sheet in a short distance—up to, say, 24 in.—has received considerable thought and effort for here would be a way to eliminate those interruptive effects that can take place during a lengthy drainage period. Several ways of accomplishing controlled formation have already been developed and more are sure to come.

One of the methods that falls into this category is the Inverform process that has been developed at St. Anne's Board Mill Co. in Bristol, England, and this report will be a brief review of the work that has been accomplished by these people.

An investigation on their part of a means to manufacture high grade folding boards at higher speeds than normal for cylinder machine operation led to the building of a 20-in. trim experimental machine. The work that was started in 1951 on this unit began to show promise with a process of upward dewatering and the experimental work was elaborated on in this direction.

Due to the success of the experimental model, in 1955 St. Anne's converted its four-cylinder no. 4 machine to a 140-in. fourdrinier with four Inverform units. Walker Bros. Ltd. built the equipment, working in close cooperation with St. Anne's engineers and papermakers.

In the basic arrangement of this machine, there are four Inverform units which are alike and each has a very simple flowbox ahead of the top wire. The top wires are 40 ft. long and the bottom wire is 200 ft. long. Stock flows onto the bottom wire immediately before the nip created by the top wire passing around a large diameter forming roll. Two large diameter table rolls support the bottom wire under the forming roll.

At the first ply forming position of a multiply machine, a certain quantity of water will naturally pass downward. However, after the first ply has been formed, very little water from the following plies can pass downward and must therefore be extracted upward.

The sheet is formed in the nip of the forming roll and the two wires with the stock between them, passes into the auto-slice position. The original auto-slice arrangement incorporated a brush roll assembly to facilitate the travel of the extractable water up an incline to a slice collector. All of the brush roll assemblies have since been replaced by suction slice arrangements with the leading edge of the slice slightly depressed into the top wire.

Conventional flat boxes are used on the bottom wire in the first Inverform position only. Inverted suction boxes are used in the second and subsequent ply arrangements following the auto-slice.

The sheet is then pressed by a rubber-covered baby press roll with the bottom roll being built into a suction box. The top roll is fitted with a suction slice. All wire runs incorporate automatic guiding and tensioning devices and they are all oscillated. The top wire frames are hinged at one end and can be lifted hydraulically in a matter of seconds.

The complete wire part is driven by an electric drive using magnetic amplifiers, with the forward drive roll as the master section; and the couch, breast roll, and top baby press roll in each wire having helper motors. Other possible drive arrangements are under consideration at this time.

The total vacuum requirements for the complete wet end are extremely low as none of the boxes pull over $3^1/_2$ in. of mercury.

All wires on the machine are sewed in position. The actual time to silver solder the wires is approximately 20 min. per wire.

Normal production runs for St. Anne's has been on white lined chip, white lined manila, and body board for coating, with a basis weight range between 45 and 80 lb. per 1000 sq. ft.

At the time of the rebuild on this machine, only the wet end was changed. The existing stack driers pose a very definite limitation to the machine capabilities and a program is under way at this moment to revamp the complete dry end.

Some typical production runs are as follows, but you must consider the fact that all speeds are predicated on drier capacity as opposed to formation potential:

54 lb./1000 sq. ft.—bleach lined body board—330 f.p.m.
70/1000 sq. ft.—white lined chip board—250 f.p.m.

Derry Akeroyd, Sales Engineer, Beloit Iron Works, Beloit, Wis.

The writer has seen a run on 0.018 caliper white patent coated board made at 500 f.p.m. which was at the limit of the present wet end drive. Because of the drier capacity problem, the sheet was dumped in the press section. It can be stated definitely that the sheet at this higher speed looked better than that produced on conventional vat machines at slower speeds.

Strength characteristics of the board made on the Inverform machine compare favorably with that made on a normal cylinder machine. Tensile ratios, in the neighborhood of 2.7 to 1, are a definite improvement. The sheet is more level and formation at higher speeds is at least equal to and usually better than conventional operation. Ply adhesion is very much better. In addition, it has been found that wide changes in freeness have little effect in the operating efficiency of the machine.

Beloit Iron Works is very much interested in the Inverform process and has entered into an exclusive licensing agreement with the St. Anne's people which allows us to build the equipment in this country as well as a number of others.

The work that has been done on both the experimental and production machines clearly shows great promise for both plied papers and boards at speeds heretofore thought improbable. Papers have been produced at speeds in excess of 2000 p.p.m. and board at speeds in excess of 1400 p.p.m. We at Beloit feel that with the Inverform the paper industry now has one more tool available for producing improved boards and papers at higher speeds and also the possibility of developing grades heretofore unknown.

Presented at the meeting of the Northern District of the Empire State Section of TAPPI held in Watertown, N. Y., Nov. 8, 1959.

New England Section

The following paper was presented at the October 2-3 meeting of the New England Section.

Some Problems in Printing Boxboard

James H. Wing

THE initial statement to be made when discussing "Some Problems in Printing Boxboard" is that there certainly are some problems in printing boxboard. These problems range from ink, equipment, personnel, operating procedures, costs, customers, to boxboard. This discussion will confine itself to boxboard and its relationship to printing in theory and practice. Even this vast subject will be extremely condensed to three selected problems which the writer feels the technical boardmaker may be able to do something about.

JAMES H. WING, Group Leader, Research & Development Dept., Robert Gair Paper Products Group, Boxboard & Folding Carton Div., Continental Can Co., Inc., Uncasville, Conn.

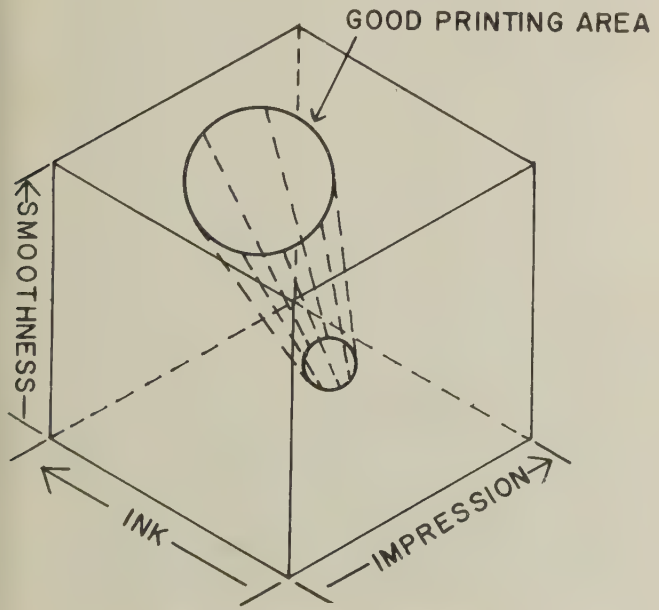

Fig. 1. The cube of printing

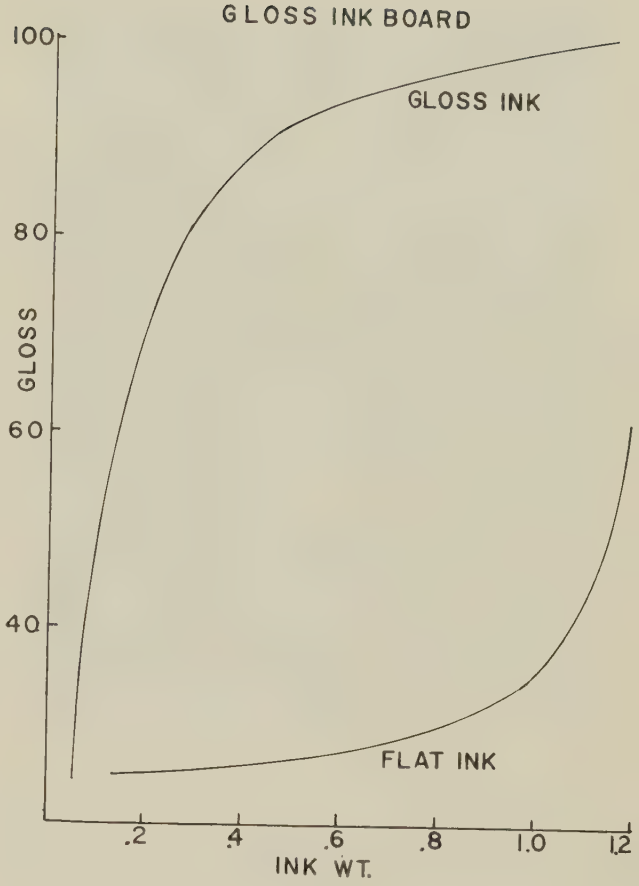

Fig. 2

Also, it must be defined now that this information is directed toward letterpress printing. Approximately 80 to 90% of the current boxboard conversion is done with letterpress equipment and therefore it seems wise to restrict the confines of this information to letterpress printing. Some of the information may be extrapolated to include other printing processes.

In theory, the printer obtains correct ink weight and impression by system which is outlined in Fig. 1. This is called the cube of printing. One side of the base of the cube illustrates an increase in impression, and the other side illustrates the ink quantity applied to the boxboard. The third dimension of the cube illustrates the smoothness of the boxboard.

Now, let us discuss each of these variables. Impression is the amount of squeeze necessary to subject the boxboard to in order to accomplish the end result of good contact with the sheet of boxboard. This squeeze or impression may vary anywhere from 3 to 15 thousandths of an inch depending on the board and the particular copy to be printed. The printer must choose some impression value that will be suitable.

Ink weight is the amount of ink applied to the board surface to make a satisfactory job. This may vary from 0.40 lb. per 100,000 sq. in. to 1.0 lb. or more per 100,000 sq. in. The weight of ink necessary to produce a satisfactory job will depend on the specific gravity of the ink and the boxboard surface. The printer must choose some weight of ink that will give the desired results.

Smoothness is that elusive characteristic of boxboard that may possibly be measured by taking a picture of it, leaking

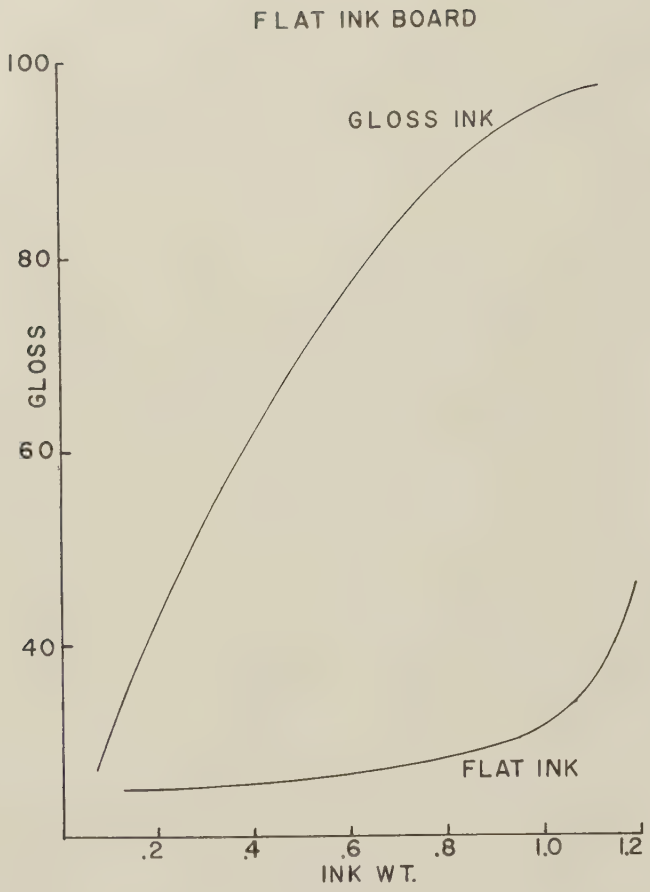

Fig. 3

air out over it, running an educated stylus over it, or by viewing it with the well-known impartial observer. Anyway, it is there and has various degrees of its character from good to impossible.

Now, let us return to the cube of printing and analyze this theoretical description of some of the things the practical printer must contend with although it is doubtful he has ever

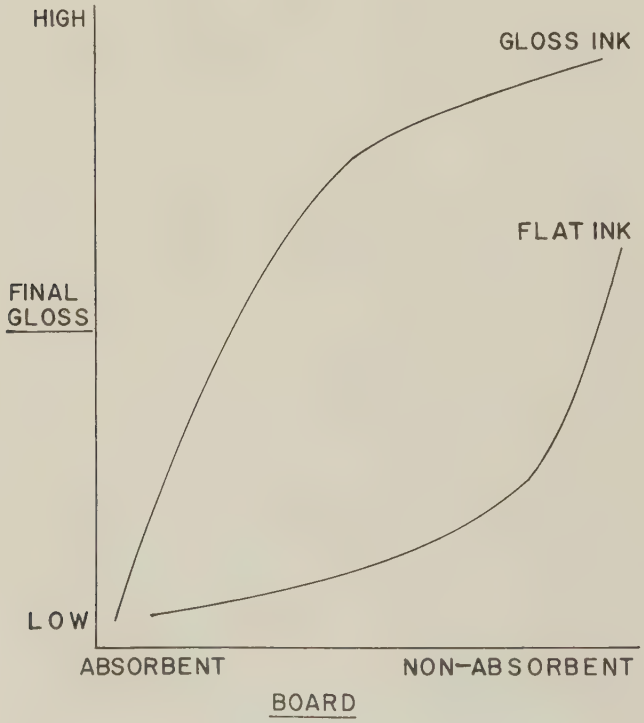

Fig. 4. Final gloss of printed surface

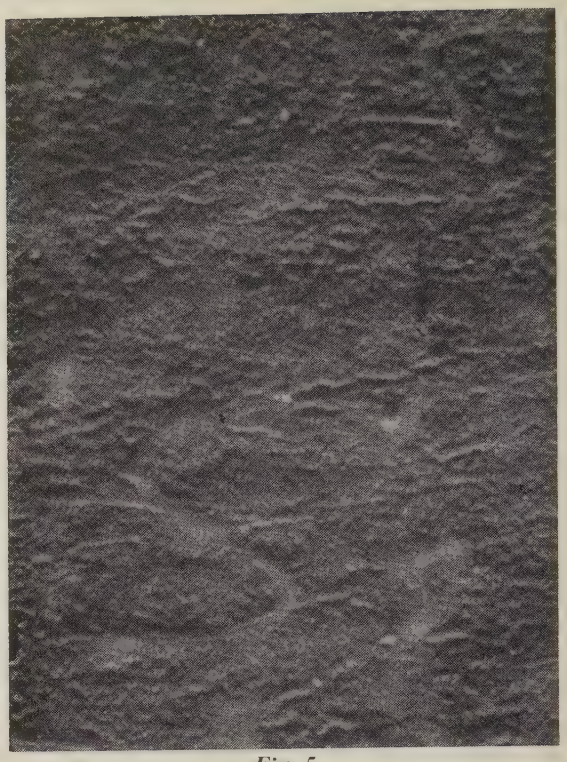

Fig. 5

thought of them in these terms. In the center of this cube will be seen a truncated cone and it is the writer's concept that the volume of the cone be called the good printing volume. In other words, in any situation, if the printer can stay within the confines of this cone, he will obtain good printing. With any board of a given smoothness, the printer will have a plane area of selections of impression and ink weight combinations from which to print his boxboard. The variations in caliper of the boxboard will take up some impression selection and the quantity of ink transfer variable will take up some of the ink weight selections. The printer tries to adjust to the center of the area and hopes the board variations of caliper and ink transfer are not so great as to put him outside the area of good quality printing.

If the impression is too light, full coverage will not be obtained and if the impression is excessive, full coverage will be obtained as long as sufficient ink is available but the defect known as embossing will occur. If the ink weight is too light, full coverage will not be obtained as in the same manner as with light impression. If the ink weight is too heavy, the defect known as offset will occur. So, for any given boxboard smoothness, the printer must choose a combination of impression and ink weight that will give quality printing.

Now, let us follow the smoothness variable and it will be seen that the area of selection for ink weight and impression is smaller and a high value of ink weight and impression is necessary when the board is less smooth. As the smoothness increases, the area of selection increases and tends toward the lower values of ink weight and impression. So, this would lead one to feel that the smoother the board the easier it is for the printer to make his selection of impression and ink weight to arrive at quality printing.

In practice, the printer depends on experience and trial and error to arrive at quality printing. The printer views his boxboard supply and judges it for smoothness in terms of packing (impression) and notches (ink weight). The actual impression and ink weight are not arrived at until a few hundred sheets have been run. It is only then that he will be printing with his combination of impression and ink weight on a board of given smoothness. As long as the boxboard's characteristics do not change, the printer can use his selected

combination and obtain printing quality consistent with the boxboard.

The second problem is the problem of obtaining the proper degree of gloss. How does the printer obtain the desired gloss?

Let us look at Fig. 2 which depicts the effect of ink weight on final gloss. In this particular case, we are considering a gloss ink board with two different inks on it. The gloss ink obtains a high degree of gloss after the ink weight is increased slightly whereas the flat ink tends to rise very slowly in gloss until it reaches a fairly high ink weight and the gloss rises rapidly. The gloss ink behaves quite nicely with weight variation once the maximum plateau has been obtained.

Figure 3 is the consideration of a flat ink board with the same two inks and in this case the gloss ink has difficulty in obtaining a high degree of gloss whereas the flat ink can behave quite uniformly over a wide range of weights.

In Fig. 4 the effect of board is illustrated. The x-axis indicates board of increasing ink holdout. The various boards have been printed with the same weight of either gloss or flat ink. As may be observed, there is very little gloss obtainable from either ink on the very absorbent board, but as the ink holdout increases, the gloss ink has a high degree of gloss whereas the flat ink remains flat until the board becomes so nonabsorbent that even the flat ink develops a high degree of gloss.

It can be seen that the factors affecting final gloss of a printed surface are type of ink, board characteristics, and ink weight. The printer can obtain the desired gloss by using the correct combination of the above three factors.

In practice, the printer requests a board of a known absorptivity and asks the ink maker to provide him with an ink to give a certain gloss on the board. The printer can only adjust the ink weight to control gloss. As long as the absorptivity of the boxboard does not change, the printed surface will gloss correctly.

The third problem is the problem of small area mottle. By small area mottle we mean the characteristic lack of uniformity of color of pastel shades of ink on coated boxboard. This defect will occur on uncoated boxboard but is much more prevalent on coated boxboard and the reason for this will become apparent.

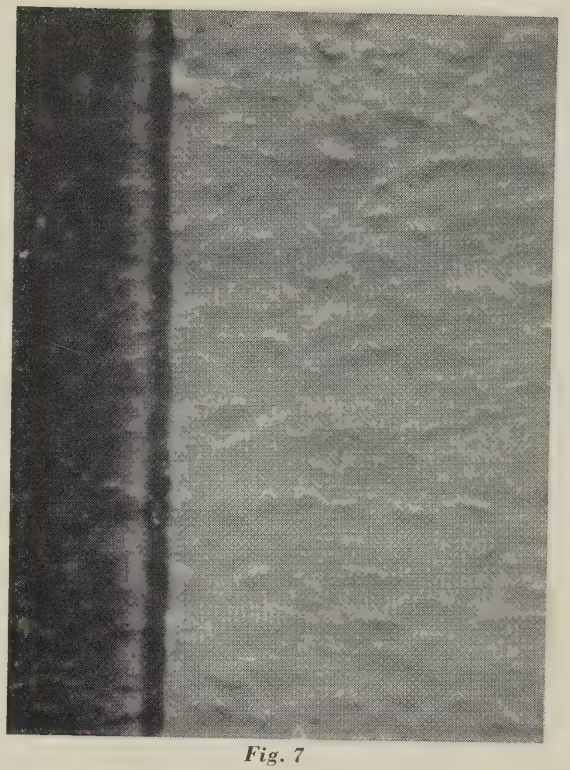

Fig. 7

If any of you were to accompany the author to the nearest supermarket or drug store, a number of examples of this defect could be obtained readily. While you, as purchaser of the packaged item, might not be highly impressed with this defect that will soon land in your waste basket, the purchasers of the printed boxboard from the printer and the printer himself is concerned and you as technical boxboard makers should be concerned because it is an evidence of lack of quality.

Figure 5 illustrates small area mottle. In this particular case it is a clay coated boxboard printed with a light blue ink.

In theory, this defect is caused by the ink squeezing off from the hard high spots of the clay coating and flowing into

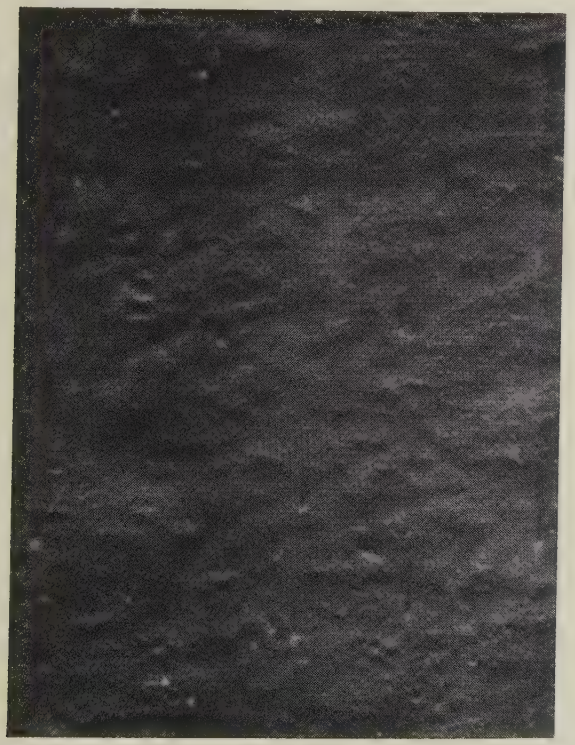

Fig. 6

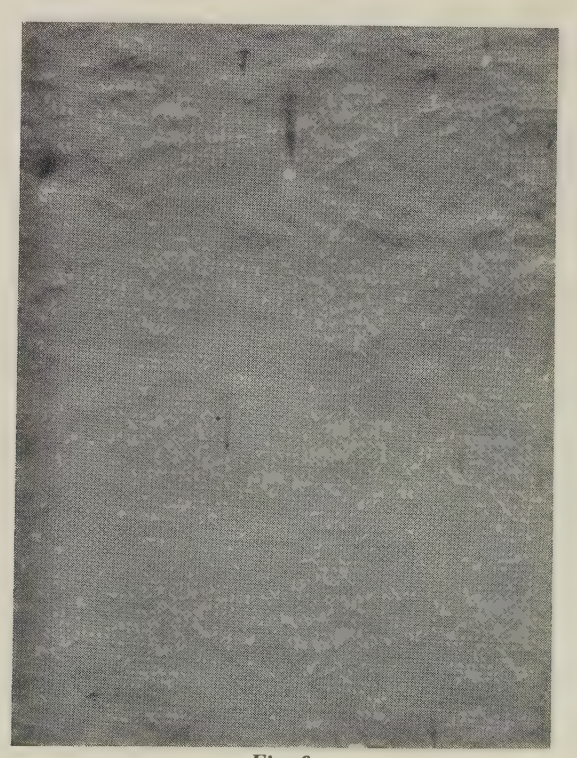

Fig. 8

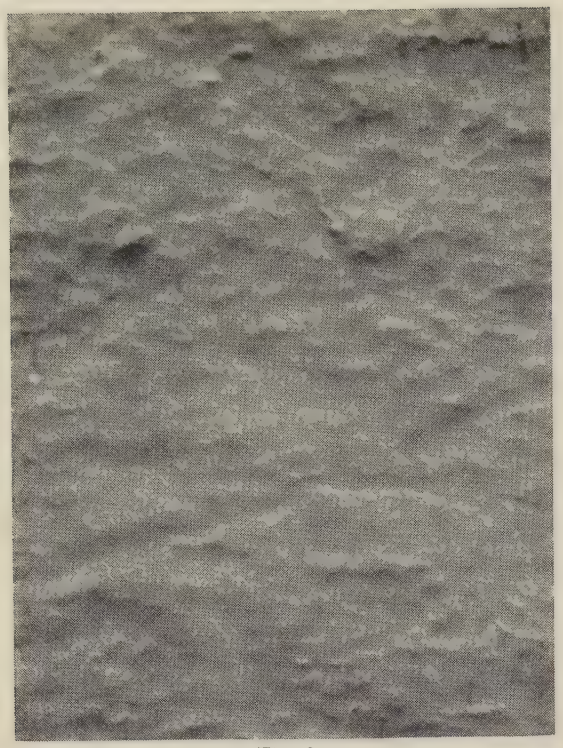

Fig. 9

the valleys or low areas. In other words, an excessive pressure has been built up at the hard high spots which have to assume the entire force of the impression. This force is sufficient to force most of the ink from the hard high spots to the low areas.

The quantity of low opacity ink left on the high spots is insufficient to give adequate depth of color at the high spots, and the situation is exaggerated because ink flows down into the low area to add to any ink already present. This double ink film will have a greater depth of color thus creating the optical impression that the small remaining color on the high spots is even more lacking in color. To illustrate the effect of color density, Fig. 6 shows this same board printed with a red ink. While the mottle is still present, it is not as noticeable as with the other ink due to color density.

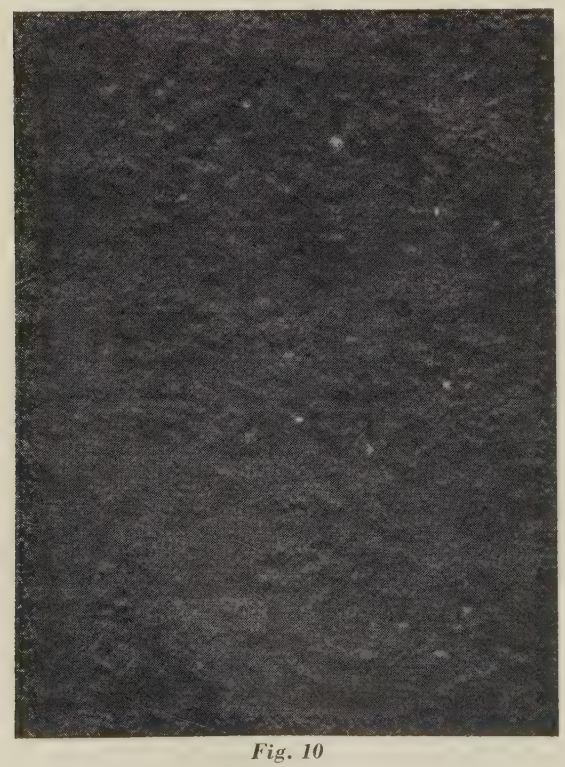

Fig. 10

Small area mottle is quite similar to the edge effect which is illustrated in Fig. 7 and is very common to letterpress printing. The edge effect is a ridge of ink on the edge of the copy with an adjacent line of little or no ink next to it. The ink in this case has squeezed out from under the printing plate.

Another way of considering small area mottle is that it is similar to printing from a screen plate in the sense that the roughness of the boxboard serves as a crude screen plate in reverse.

If the hard high spots which cause this defect were to have a softness to them or to have the ability to immobilize the ink, the possibility of obtaining a satisfactory print with sufficient impression would be greatly increased. Of course, the possibility of obtaining softness to the high spots is somewhat improbable if one will consider the calendering operation. So, that leaves the coating formulation as a possible, but doubtful method of correcting ink flow from the high spots. The obvious answer is to make a smooth coating such as shown in Fig. 8 as compared to the rough coating in Fig. 9, which is the coating for the small area mottle depicted in Fig. 5 which was the original picture of mottle.

Figure 10 illustrates the same low opacity blue ink printed on the board depicted in Fig. 7. As can be seen, the small area mottle is very much reduced because there are no hard high spots for the ink to squeeze off from.

In actual practice the printer can do little about correcting this defect. It can be improved and perhaps eliminated depending on the seriousness of it. He can change his printing methods such as speeding up or slowing down, increasing or decreasing impression, more or less ink. He can alter his inks so that they will not flow as readily by vehicle changes or by the addition of magnesia. He can build more opacity into his inks if possible. Perhaps he can use rubber plates instead of metal. These are all practical cures to a problem that is actually corrected only by the proper board.

In conclusion of this consideration of some problem in printing boxboard, it must be said that the printer has been able, through his own skill, to remedy many of the unnecessary defects common to boxboard. The term "unnecessary defects" has been deliberately used here to emphasize the thought that proper boxboard making technology can and will eliminate these defects so the printer of boxboard will not have to spend his skill on correcting such defects. As a final statement in this conclusion, it must be said that these and many other problems in printing boxboard would be lessened if those making boxboard, whether good or bad, would make it uniform.

Presented at the meeting of the New England Section of TAPPI held in Great Barrington, Mass., Oct. 2–3, 1959.

Eleventh Coating Conference

of the

Technical Association

of the

Pulp and Paper Industry

Edgewater Beach Hotel,

Chicago, Ill.

May 23–25, 1960

Doctoring the World's Largest Paper Machine

HAROLD E. DUNLAP

THE world's largest paper machine, a new 342-in. Black-Clawson newsprint installation at Fort William, Ont., is producing 32-lb. newsprint for Great Lakes Paper Co. at the rate of 500 to 2250 f.p.m., equivalent to a maximum production rate of 420 tons per 24 hr. Because of its size, every component represents a deviation from a "standard" installation and required design modifications; this was true particularly of parts such as doctors.

Black-Clawson called in Lodding Engineering Corp. on the problem of doctor design and installation, and Lodding together with its Canadian sales representative, Pulp and Paper Mill Accessories, Ltd., of Montreal, undertook the assignment.

Figure 1 explains the terminology used in describing doctors and illustrates the conventional mounting of a doctor back.

Design and fabrication of doctors have been evolved empirically through the years, principally for want of more satisfactory approaches. No simple formula was considered applicable because so many design considerations are involved which, experience has shown, are more important than face width alone. Material selection, blade angle, blade pressure, journal location, mounting arrangements, and oscillation are all elements which depend on roll surface and service conditions.

Now that 340-in. doctors are in actual operation, it is a temptation to follow their development in reverse by using the Monday morning quarterback technique and saying it was planned that way. Actually, past specialized experience with many hundreds of doctor applications was invaluable in arriving at a new method of design. The mathematics involved were incidental by comparison.

In doctor design, the traditional starting point has been the determination of the minimum moment of inertia required. Since the blade must be in contact with the roll at all times, and at reasonably constant blade loading, rigidity is essential for all sections. The ultimate rigidity is determined by the doctor backs which, if considered as freely supported beams, are acted upon by externally applied loads and the fundamental vibration frequencies of the sections. Provision for controlled oscillation must be considered too, so three-dimensional stress analysis is indicated at this point, since only a few special cases involving six stress components have been solved, however, this possible approach was not pursued.

A basic mechanical engineering principle states that a uniformly-loaded beam simply supported at both ends will have maximum deflection at its center. It can be reasoned that if a short doctor operates satisfactorily, it is apparent that its deflection is within acceptable limits and experience makes it safe to assume that a longer doctor could tolerate this same deflection. This concept might be called design by extrapolation or, to make it sound more high-powered, Lodding's law.

HAROLD E. DUNLAP, Chief Draftsman, Lodding Engineering Corp., Auburn Mass.

We need concern ourselves only with simple mathematics to arrive at numbers useful for design purposes because first, we must assume we have a uniform load (if it is impossible to calculate the cumulative force due to random and unknown deflections); and second, we assumed arbitrarily that we were dealing with first mode natural sectional frequencies of 10 c.p.s. or higher.

The deflection formula for beams supported at both ends with uniform load gave us the answers we needed:

$$\frac{5\,WL^3}{384\,EI} = d = d' = \frac{5\,W'L'^3}{384\,E'I'}$$

In this formula "W" represents the total force developed from short impact and friction between blade and roll and the formula then boils down to what we assumed: the force acting on a 270-in. blade is directly proportional to that acting on a 340-in. blade. Assuming the same modulus of elasticity for each blade and solving for I' gives a factor of 2.5 I as the relative moment of inertia of the 340-in. doctor necessary to maintain the same degree of stiffness the 270-in. doctor has.

At this point the final figures needed by the design engineer depend on vibration data. All beams respond to any sort of impact, their displacement cycle depending on the stiffness of the section. We believed that any attempt at a complete vibration analysis would result only in time wasted, principally because so many unknown forces were involved. Damping

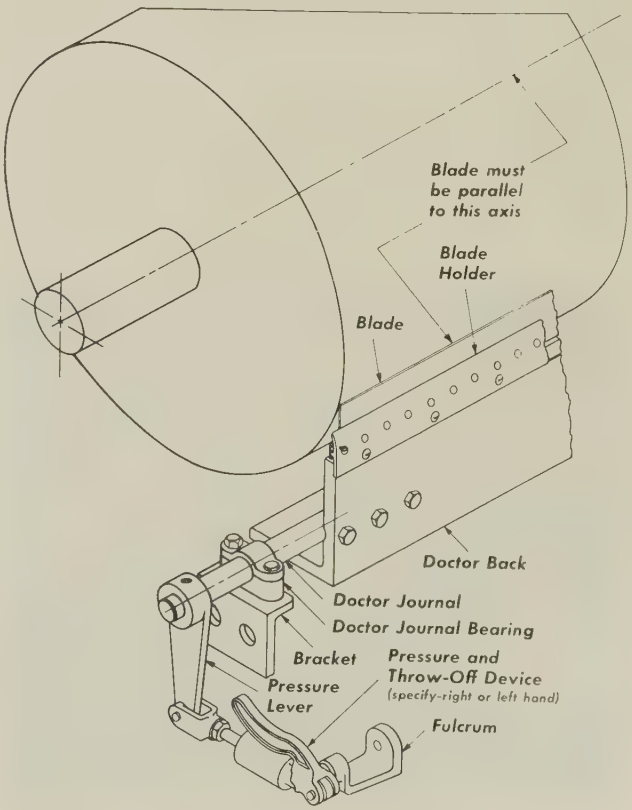

Fig. 1

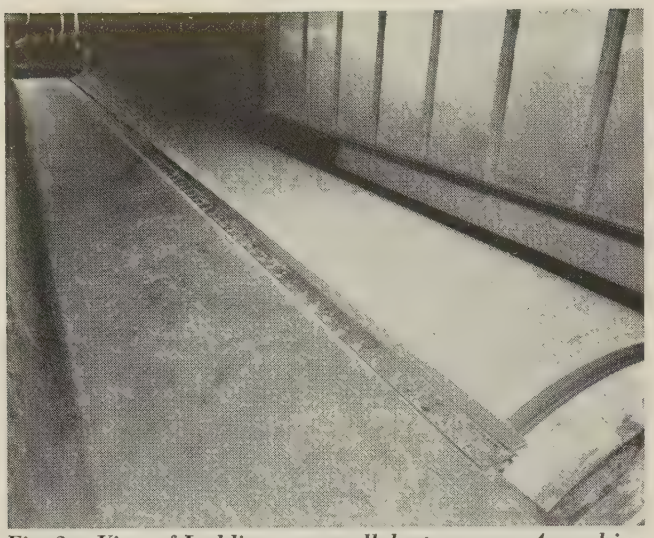

Fig. 2. View of Lodding press roll doctor on no. 4 machine at Great Lakes Paper Co., Fort William, Ont., taken while machine was idle

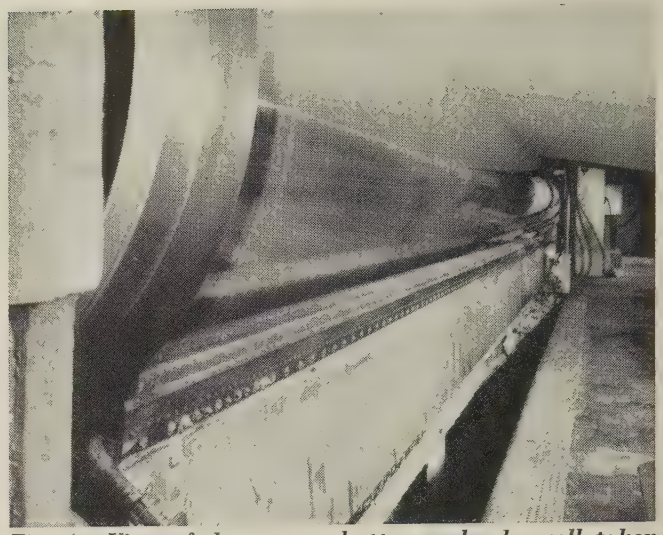

Fig. 4. View of doctors on bottom calender roll taken while machine was running

speed, for example, depends on the natural frequency of a beam; well-designed doctors are not unduly disturbed by random impact imparted by the sheet or deposits on the roll being doctored, but complications do develop if the frequency of transmitted vibrations is sympathetic with the natural vibration frequency of the doctor.

Prior to designing these doctors, several field trips were taken to various mills. Equipped with a vibration meter and pick-ups, an oscilloscope, oscilloscope camera, and a stroboscope, checks were made in an effort to determine the frequency and amplitude of vibration present in existing doctor installations, machine bearing, machine frames, and machine room floors.

The vibration meter alone was not too useful as it measures average velocity, acceleration, and displacement from which average frequency must be calculated. The oscilloscope, however, was exceedingly valuable; its screen indicated the actual displacement and shape of the wave. Often the wave shape indicated the effect of two or more forms of vibration or a relatively high frequency wave superimposed on lower frequeny waves.

Records were made of data observed and pictures were taken of the oscilloscope screen when a particularly interesting wave form appeared. Pictures were also useful to stop a moving wave form so that its shape and displacement could be studied visually.

Full investigation of paper mill vibration, or even that present on doctor backs, could easily become a lifetime work. For our immediate purposes, however, it proved that low frequency vibration was the most predominant in the vicinity of paper machines. We arbitrarily decided, therefore, to design our doctor backs to have a first mode natural frequency of 10 c.p.s. or higher. This figure, incidentally, was higher than any anticipated roll speed in revolutions per second.

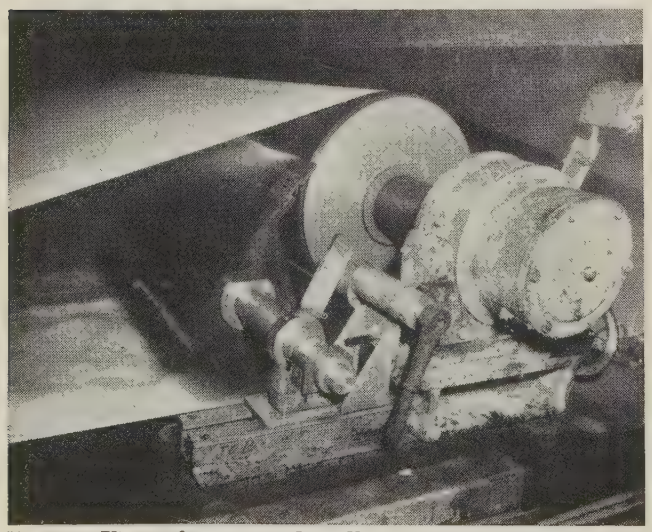

Fig. 3. View of auto guide roll doctor no. 4 taken while machine was running

First mode, or primary, natural frequency can be expressed in simplified form as:

$$f = \frac{3.13}{\sqrt{d}}, \qquad \text{or} \qquad d = \left(\frac{3.13}{f}\right)^2$$

by substitution

$$d = \left(\frac{3.13}{10}\right)^2 = 0.098$$

This figure represents the maximum allowable deflection to achieve our goal of 10 c.p.s. or higher.

Substituting in the original deflection formula given above, the desired weight in pounds of each section to be designed was found to be 5.5 times the moment of inertia of each section

DESIGN

As a starter, the foregoing theoretical design procedure was applied to standard section short face doctors. When these figures were projected to cover known characteristics of 240- to 270-in. doctors, the results agreed so closely that it seemed safe to apply the same procedure to the design of 340 in. blades. It helped greatly, of course, to be able to depend on long established general principles of good doctor design including selection of blade materials, blade angle and pressure, journal location, external loading methods, and the like. Each individual application was laid out to include the space limiting factors involved. Desired location of the doctor blades on the various rolls was established and due consideration was given to path of the sheet during start-ups or breaks as well as during its normal run.

By trial and error, suitable sections were then established to conform to these space restrictions. Meanwhile, checks were constantly being made to assure that the ratios for moments of inertia and weights with respect to the base doctor design selected for each individual application were being maintained.

Eventually, a number of tentative section designs were established for all applications involved. These tentative designs then became the object of many hours of discussion

between engineering and production departments. Each phase of the fabricating, stress relieving, and machining operations was thoroughly worked out. In a number of cases, it was thought that the tentative designs could be altered to better balance the continuous bead welds involved or to provide more convenient locating points and clamping surface for the structural components of each section. Designs were then reworked to conform to these decisions with some compromises made, as necessary, to maintain the basic moment of inertia and weight relationships previously determined.

After fabrication of these sections was complete, several were placed on a mock-up floor erection where mounting conditions similar to those on the paper machine were duplicated as closely as possible. A shaft-mounted eccentric weight was then mounted on bearings at various points of each doctor back. This eccentric weight was then rotated by a direct current motor drive with varying speeds.

Displacements occurring from the resulting forced vibration were sensed by strain gages placed at mid and quarter points on the section and were read on an oscilloscope. The results were highly satisfactory as first mode resonance by actual tests varied less than 15% in all cases from the admittedly rough calculations made during the design stage. These checks also established the frequencies of the second and third modes of vibration which, of course, are far higher than frequencies expected in doctoring service on the paper machine.

The paper machine for which these doctors have been designed has now been in operation over 6 months. At last report, paper was being produced at approximately three-quarters the maximum machine design speed. The doctors have fulfilled their various functions well. Direct questioning of operating personnel have failed to provoke any unfavorable comment or to indicate any inadequacy in the various section designs.

It is entirely reasonable that many beam problems may be solved using somewhat similar systems. Perhaps other applications would necessitate differing premises and evaluation of forces likely to be encountered as well as the use of other deflection formulas. The performance of this equipment justifies the several premises made, and the reasoning followed using the design by projection method. The success of these doctors has proven its value as a design tool.

This "new" approach to doctor design probably appears unscientific in an age when so few things remain unpredictable or unmeasurable. It is justified for the best of reasons . . . it works.

A Rational Approach to Aeration Methods Involved in Backwater Solids Recovery

L. L. KLINGER

Aeration methods involved in the recovery of backwater solids are common in the progressive paper and paperboard mills. However, certain factors entering into the selection should be evaluated carefully in order to achieve the best performance. Suggestions and precautions are herein enumerated.

THE purpose as well as the definition of a saveall should be considered carefully. It would seem that a common connotation attached to a saveall is more in the nature of a "catch-all." Poor mill conditions certainly can be improved with recovery units, but at best should be studied from the value of their own containment. After such a study, then unrecoverable fiber and filler should be handled with some sort of a saveall.

A previous paper by the writer (*2*), delivered for a waste conference represented by other industries besides the paper industry, dealt with some data covering a paper machine water balance. Figures were also given on compiled saveall data.

TYPES OF SAVEALLS

In general today, three types of modern savealls are used to handle paper mill white water. These are as follows:

1. The sedimentation type, such as used in cold process water treatment, wherein solids are removed at the bottom of the unit and the overflow consists of clarified water. This type usually requires chemical coagulants and adjuncts to operate successfully.
2. The vacuum saveall type, or the rotary drum type, either working off an atmospheric leg or vacuum pump. There are various modifications, but in general this type of machine is applicable where drainage characteristics of the stock are fairly free.
3. The flotation type, to be dealt with in this paper.

The first type, or sedimentation type, has been used extensively in the past. Its great size in the form of rectangular tanks under the machines with bottom scrapers to reclaiming pits is well known to papermakers, especially those who have been in the business two or three decades, or who are now working in the older mills. The limitations of these older units certainly created serious thought toward more efficient and more compact types of units.

Today the sedimentation unit has been greatly improved, but, even so, the concept relative to its usage has also changed. More likely today and in the future, its greatest potentiality lies in the field of second-pass operation wherein overflow from other savealls are collected. These wastes are then treated for acceptable discharge to the river, or are sent to secondary treatment for further B.O.D. removal.

The second type, or vacuum saveall type, has had considerable success in the reclamation of the freer and more virgin stocks. The fibrous mat formed on the saveall wire is quite easily handled for return to the machine or stock preparation system. When sheets are heavily loaded or contain fine coating materials, the filter leaf test should be used to determine the practicability of the vacuum saveall. The filter leaf test is quite accurate and is conducted as an adjunct for specifications by reputable manufacturers.

So much for the background into the sedimentation and vacuum saveall. They were discussed briefly because, when judiciously chosen, they are doing a banner job and certainly should be considered in any evaluation covering new or major programs.

CONVENTIONAL STUDIES OF THE FLOTATION PRINCIPLE

The principle of the flotation saveall is that of continuously dissolving air under pressure into a continuously collected excess of white water. The result is a flotable material consisting of fiber and filler. By adding certain chemical flocculants to the whitewater in small amounts a greater air buoyancy is provided. Then by releasing the total mass to the atmosphere in some sort of container, air bubbles carry fiber and filler in the whitewater to the surface. This principle should be referred to the laboratory for their investigation.

Any well-equipped routine laboratory can conduct a thorough investigation for engineering and management which should be quite enlightening as to the prospects of the flotation principle.

Berman & Osterman report on a fairly well established and

L. L. KLINGER, Technical Assistant to the President, Whippany Paper Board Co., Inc., Whippany, N. J.

accepted method (*1*) which is simply to plot rise rate of solids in white water as a time function versus per cent subnatant water as a persistance function. They obtain data for plotting the curve as follows:

1. Establish coagulant dosage by conventional qualitative and quantitative flocculation tests.

2. Pressurize samples of white water treated with the optimum chemical dosage in a test pressurization cell. Pressure is raised in the cell by introducing compressed air until the desired pressure is obtained. The contents of the cell are agitated to insure that saturation of air is achieved.

3. Pressure is released as soon as aeration in test cell has reached the desired level. Contents of the test cell are then transferred to a calibrated float chamber, such as a one liter graduated cylinder.

4. Timing at increments by means of a stop watch the per cent of subnatant water. Readings of this type are recorded for 30 min. or more until no substantial increase in subnatant is observed.

5. Plotting of time as the abscissa and subnatant as the ordinate will result in a curve through the selected points.

The curves they show are quite typical in that the per cent subnatant increases quite rapidly in the initial few minutes; reaches an equilibrium and then normally levels off.

By repeating the test, with varied air pressure, and chemicals, it is possible to predict:

1. The flotation air requirements and the operating pressures of a full scale operation.

2. The design area and dwell time of the flotation chamber for a full scale operation.

3. The performance reproducibility and efficiency for a full scale operation.

The foregoing tests have been conducted in a much simpler manner by the writer. First the normal coagulation tests are made. If these samples treated with proper chemicals are then violently shaken to aerate them, it can be predicted by the relative rapidity of the rise of the flotable materials and the turbidity of the subnatant what can be expected in the way of flotation efficiency. Of course, any such tests become useful only after experience in operation of commercially sized savealls.

If these tests prove to be of any significance to those interested in the flotation principle, there are pilot plants available from reliable vendors who will be glad to work with those parties. By actually removing small quantities of the waste continuously and using the pilot plant for the study, a much clearer and better understanding can be derived. Test results can then be extrapolated into commercial units, and sizing and pricing becomes quite realistic.

VARIETY OF COMMERCIAL UNITS

Originally the flotation saveall came from Europe. The name Sveen-Pedersen has become synonymous in the trade with flotation. Historically, as is commonly known, Nils Pedersen, a Norwegian engineer, conceived the idea of flotation. Karl Sveen promoted the idea of the colloidally active glue as a flocculant adjunct. The colloidal aid and the saveall combined into a harmonious union which in many instances cannot be surpassed even to this day. The Sveen Pedersen saveall was introduced into the United States about 1937.

Today, two companies are competitive in the promotion of the Sveen-Pedersen principle in more or less its original form. These are the Sveen-Pedersen Sales Corp. and the Permutit Co., the latter recently acquiring the rights of the Bulkley-Dunton Co.

Since the original Sveen-Pedersen, various competitive units have been introduced, some with comparatively radical new design, and others more in the nature of adaptation of more ideal hydraulics.

One of the earlier patented units, the Adka saveall, incorporated a vacuum principle. In this unit, the whitewater was thoroughly aerated at atmospheric pressure. It was then drawn by vacuum into a large mushroom shaped tank and held under vacuum. Dissolved air then left the whitewater and the small bubbles attached themselves to flocculated material which then rose to the top. Skimmers removed these solids. To the writer's knowledge, this unit is no longer offered because of certain difficulties in its operation which were never completely overcome in the general overall range necessary for a real sales success.

The Sveen-Pedersen Type of Saveall

The Sveen-Pedersen type of saveall will be described quite fully as it has had wider acceptance than the more recent and competitive types because of its tenure in America. Also a fuller description of it will suggest the principle involved in the competitive units.

Figure 1 shows a typical flow sheet for a Sveen-Pedersen type flotation saveall. It also for practical purposes serves as a typical elevation. White water to be clarified enters a collection tank of suitable size, usually having about five minutes retention time. It is pumped by means of a high pressure centrifugal feed pump at 3 to 4 atm. into an air retention chamber suitably baffled. About 1% atmospheric air is metered through a rotameter by an air injector operating

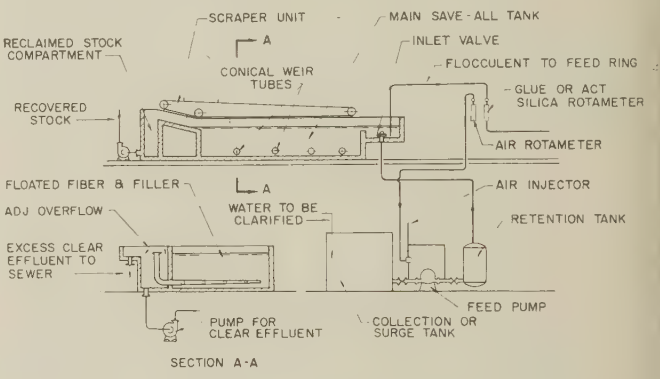

Fig. 1. Sveen-Pedersen type of flotation saveall

from the pump and is drawn into the suction of the pump. By means of the high pressure and the retention of about one minute in the tank, air is finely dispersed and occluded around the white water particles and may be considered partially dissolved.

The aerated white water, after passing through the retention tank, by virtue of its residual pressure flows through a suitably sized pipe to the inlet of the saveall proper. At a point, just ahead of the saveall, not shown in this drawing, usually a throttling gate valve is used to reduce the somewhat excessive pressures to the patented valve in the saveall.

The patented valve is a double disk type. The water flows into a connection at the bottom of the valve and into a vertical aperture. It then discharges as a fine jet through a final circumferential orifice into the inlet of the saveall. Larger savealls may have two or three such valves. The patented valve is adjustable and is usually opened far enough to produce an audible rumble. By making adjustments between the influent throttling gate valve and this patented disk valve, a fine degree of performance can be achieved. Colloidal glue and other flocculants are usually admitted near the valve through rotameters.

As can be expected, the fine jet of white water emerging from the orifice to the atmosphere has the effect of reducing air bubblets to a minimum size and creating an occlusion of these minute bubblets around the pulp and filler particles, thus floating them.

In appearance, the solids resemble a frothy milklike

mat of foam. The greater mass of water being relieved of its solids overflows the weir and thence proceeds through the saveall.

A series of paddle scrapers drawn on a chain and driven by a variable speed fractional horsepower drive, push the floating solids across the saveall and over an inclined baffle board into a reclaimed solids chamber. These scrapers are about 6 ft. apart and travel at a rate of about 6 f.p.m. The reclaimed solids usually have a concentration of 1 to 3%.

At this time, it should be stated that the flotation chamber is a relatively long narrow rectangular chamber, quite shallow in depth, having about one quarter hour retention. Looking at the cross section A-A, the weir tube draw-off to a side chamber is shown. This drawing shows four such tubes although there are usually considerably more. These weir tubes have receiving orifices drilled and properly spaced to receive the clarified subnatant water which flows into them uniformly and thence upward to the up-draft part of the tube into the clarified water chamber. Each of these pieces has a slip tube inside the up-draft part of the tube which is adjustable to level the saveall. By checking the relative elevations and flows out the various updraft sections it is possible to balance the saveall for the best operation, and also hold it at the proper maximum and optimum operating level.

The clarified water, after overflowing the updraft outlets of the tubes, drops into the long and narrow clarified chamber along one side of the saveall. From this chamber, a pump or

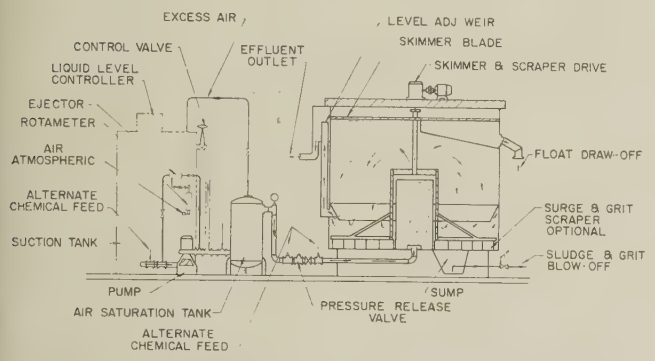

Fig. 2. Sediflotor type uses a circular design

series of pumps can transport the clarified water back for various uses in the papermaking process. During certain periods, when there is not sufficient use for clarified water, it can overflow to the sewer. However, when this happens, the clarified water is usually sufficiently devoid of solids to render it reasonably harmless to the receiving river or stream. Properly clarified water usually looks almost clear enough to drink and contains no more than 25 p.p.m. of turbidity as SiO_2. Guarantees are usually made for less than one half pound per thousand gallons or 60 p.p.m. of total filterable solids.

The reclaimed stock is pumped back from the reclaimed section of the saveall to the papermaking process where it is mixed at some convenient point with incoming stock, usually near the paper machine or at some chests preceding it.

Sediflotor Unit

Figure 2 shows the Sediflotor, sold by Inifilco, Inc. A general inspection of the operating principle reveals that it is similar to the Sveen-Pedersen previously described. White water from a receiving tank is drawn into the suction of a high pressure transfer pump operating at a pressure of one and a quarter to five atmospheres. Air is introduced by means of an injector or compressor into the suction side of the pump.

Thus the liquid waste from the pump suction tank is pressurized from the time it enters the pump until is passes through the pressure relief valve to the unit. An air saturation tank is again used, with a retention time of about 1 min. Excess air is released from the top of the tank to prevent the passage of large bubbles to the flotation unit.

From the air saturation tank, the waste passes through a pressure release valve to the flotation unit through a special distributing draft tube. The released bubbles attach themselves to the solids, or floc, and carry the material to the surface where it is removed by the rotating skimmer. Skimmer blades move the "float" into one or more discharge troughs and underflow is removed through a peripheral submerged takeoff. Heavy solids, if any, settle and are collected and removed by a scraper mechanism. A sludge and grit blow-off completes the system.

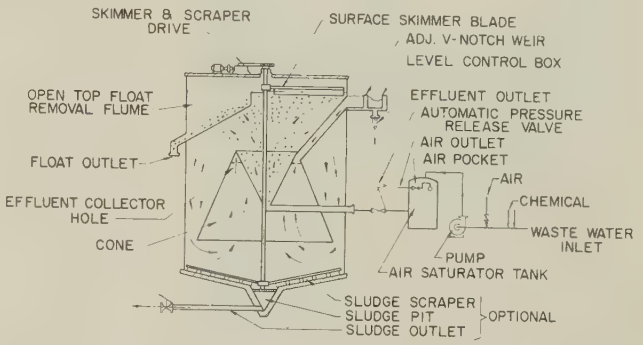

Fig. 3. The Clariflotor follows the circular hydraulic design

Since this type of unit is usually circular in design, it offers certain functional features which are not available in rectangular units. Basic up-flow hydraulics should be ideal, and removal of gritty nonflotable materials from the bottom can be handled with a rotating scraper mechanism. This gritty material is generally harmful and thus a satisfactory means of disposing of it is available.

Clariflotor Design

Figure 3 shows the Clariflotor by the Graver Water Conditioning Co. In it, incoming waste water is similarly aerated under pressure in the retention tank, such as in the previously described units and coagulants are added as may be necessary either before or after aeration.

Again, an air relief valve is used. The release of the pressure causes the air to come out of the solution in the form of tiny bubbles, the size of which depends upon the aerating pressure, the pressure drop through the relief valve, and the amount of air in solution. A somewhat different annular cone shaped distributor serves as a distribution unit for aerated water entering the unit.

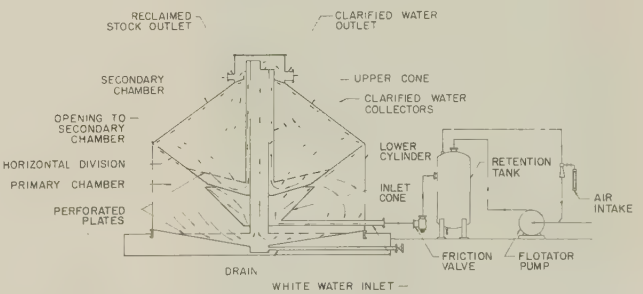

Fig. 4. The Flotator incorporates some novel design features

The air entrained suspended matter then floats to the surface where it is removed and partly dewatered by the constantly rotating skimmer blades. The clarified water rises

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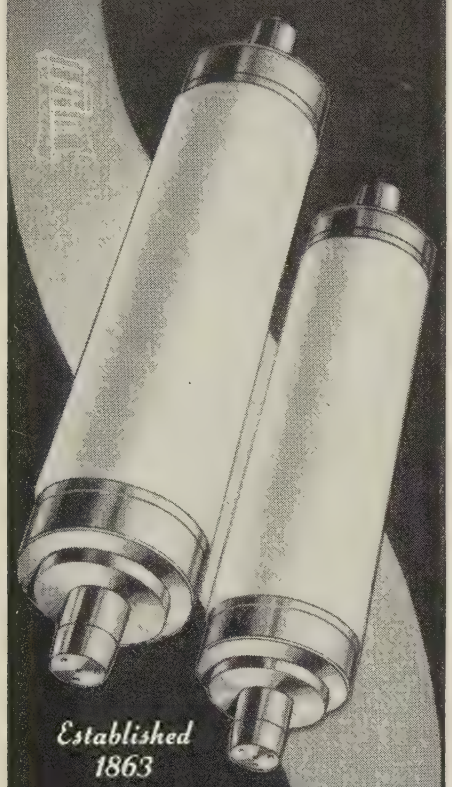

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to the collector ring at the bottom of the cone and is withdrawn from the tank for further processing or disposal. Sludge collection scrapers are also used to remove gritty, harmful material settling at the bottom.

The comments pertinent to the Infilco design concerning hydraulics and sludge removal also apply to this circular unit.

The Krofta Flotator

The Krofta Flotator is probably the most distinctive and is of a somewhat different concept when compared with the other described savealls. Figure 4 shows a cross section of this unit, usually of a circular design. Again the same kind of devices such as the feed pump, air injector, retention tank, and the pressure reducing valve are used before the saveall. Chemicals are usually added at the injector.

The aerated whitewater passes to an inverted inlet cone and thence enters the lower flotation section under the dish shaped horizontal division. The air bubbles push fiber and filler flocs under the inclined surfaces of the dish shaped division and upward toward the outer edge of the inverted dish.

From this point the floc enters the upper flotation chamber under the inverted conical surface. The aerated floc rises to the segmented annular chamber at the top of the unit and thence moves to the overflow tube for discharge.

A unique method of collecting clarified water from both the lower cylindrical part and the upper inverted cone is provided by perforated plates in each zone. The clarified water flows through each of these plates to enter the central and annular discharge tube. It is then discharged through the overflow for clarified water also located at the top of the inverted cone.

For washout in the enclosed unit, a central pipe is connected to a fresh water line. Shower frames bottom and top are fastened to this central pipe which has a mechanical joint for manual rotation when desired. This system can be cleaned simply and effectively by manual operation without opening the saveall.

Soyka (*3*) reports on the operation of such a saveall and claims these advantages: (1) half the space requirements of other flotation savealls, (2) lack of contamination due to enclosure, and (3) no power requirements for mechanical parts.

LIMITATIONS AND ECCENTRICITIES

Because of the many successful installations, it is easy to become enthusiastic over the principles of flotation savealls as used in the paper industry. However, the limitations and eccentricities of operation should be enumerated. These limitations and eccentricities are apparent only to those with considerable experience in operating such units.

The writer has had the advantage of following process design through the construction and the subsequent operation of ten such units, and through this experience can offer these comments:

1. For each particular saveall application, there exists a critical range of operation relative to chemical components of the furnish and those chemical additives necessary for the best saveall operation. Many times a fine degree of pH control is necessary and requires instrumentation.

This subject will be dealt with more extensively in the next section.

2. There is a practicable limit for influent concentration of solids to the flotation saveall, above which it is difficult to operate well, so far as overall efficiency or ratio of recovered solids to incoming solids is concerned. With too high a concentration of inlet solids, usually the clarity of the clarified water suffers tremendously because solids are not sufficiently removed. It is advantageous to divert the white water, heavily loaded with solids, back into the process rather than directing it into the saveall. Thus, only the collection of lighter waters for the saveall is then necessary. Usually such an arrangement will reduce the initial cost and size of the unit, and will keep the system as simple as possible.

3. There is a practicable minimum operating limit below which the velocities are too low to support flotable materials. If retention time is to great, the flotable materials tend to sink before they can be removed. Therefore, it is not wise to oversize beyond any anticipated future needs. It is possible to recirculate clarified water with incoming water if the saveall is oversized to accommodate future expansion, but there is the accompanying power loss to be considered. For each 100 gal. recirculated, 5 to 6 extra horsepower are expended somewhat uselessly.

4. Pump selection is important. Because air is injected into the feed pump, adjustments must be made for air in the engineering specifications. The pump for recovered solids must handle foamy, frothy, air laden solids and water, usually at very low suction heads. It usually must be specially designed with a large suction entrance, and must be close coupled to the reclaimed solids cham-

ber. Certain pumps in the trade will do the job, but many times the operation is critical. Aspirators also can be used.

5. Air-laden solids have to be put to re-use carefully in order that they do not impede the operation of the process.

6. Analysis must be made of the bacterial quality of the reclaimed white water. When containment becomes too closely controlled, accelerated bacterial, and also fungal growth, may occur which may become detrimental.

7. It was considered suitable for some time to operate savealls on fluctuating flows if a fluctuating influent existed. It is more efficient and desirable to operate with an influent-receiving tank of suitable size to level out these fluctuations. A steady, continuous flow produces the optimum results as variations increase the possibility of upsets.

8. Freedom of adjustment of the air supply is absolutely necessary. Therefore, air supply lines must be adequately designed to handle more air than may ever by anticipated. It appears that this protection tends to level out the influent metering of air, more than any other refinement.

9. Scraper adjustments are critical, more particularly in the circular units.

10. Positive and sensitive level control is imperative and cannot be underrated.

When all of these various ramifications are taken into account, it can be seen that precautions must be exercised.

CHEMICAL CONTROL

A common fallacy, following the inclination of many practical operators, is that the save all should be efficient with only slightly modified chemical control or none at all. Such ideal conditions sometimes happen, but in general are unusual.

More likely a small amount of additional flocculating agent such as alum is required. Many times this extra alum must be metered accurately such as by means of pH control requiring an electrode sampler signaling to a controller preset for an optimum pH. The controller in turn operates a small automatic valve feeding alum to the unit. Often this operation is critical—too little alum, no results, and too much alum—too heavy a floc which wants to settle instead of float.

Very seldom such units operate without a coagulant adjunct. Sveen glue is often added in the presence of alum because of a powerful flocculating effect due to the negative potential of the glue in conjunction with the positive potential of the alum. The glue, in combination with the air and flocculant, creates a condition of electropotential beneficial to flotation.

Various vendors offer formulas for preparation of colloidal glues, usually requiring a batching process.

Sometimes other coagulant adjuncts can be substituted for the commonly used glue. Activated silica and polyelectrolytes such as Separan have been employed successfully. Again, a great degree of control is necessary for the proper results both from operation and economy.

The subject of coagulant adjuncts used for flotation savealls should be the source of an excellent future paper for someone familiar and grounded with experience in the many and varied types. The field has become prolific, almost chagrining, to the papermaker not versed in all the terminology of modern chemistry.

ADVANTAGES OF THE FLOTATION SAVEALL

Some of the advantages of the flotation saveall for waste treatment are as follows: (1) Its comparatively small size and volume to complete the functional requirement of waste treatment; (2) its low cost of maintenance; (3) its low cost of installation compared with other systems, in many instances; (4) its low cost of supervision.

Of particular advantage to the papermaker are the following for a great number of various papermaking furnishes: (1) High operating efficiency, or ratio of solids leaving to solids entering; (2) utilization of fairly valuable reclaimed stock; (3) utilization of highly clarified reclaimed water; (4) reduction in stream pollution; (5) compatibility with general papermaking operations; (6) savings in chemicals; (7) savings in heat; (8) savings in water; (9) better paper machine performance and generally higher paper production speeds.

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3. Soyka, Geo. E., *Paper Trade J.* reprint (Jan. 9, 1956).

Received Oct. 16, 1959.

The writer wishes to acknowledge the use of sales brochures of the various companies whose units are described herein and especially the Graver Water Conditioning Co. for their aid in preparing the illustrations.

RECENT BOOKS

Labor-Management Relations. By Charles Wiedemann. Reinhold Publishing Corp., New York, 1959. $5^1/_4 \times 7^1/_2$, 142 pages. $3.75.

A review of labor-management relations has seldom been more timely. Although the road to harmonious relations between the protagonists of modern prosperity may be tortuous, experience has planted useful signposts along the way. Lest these become dim with time, constant inspection is required.

In pointing out the way, the author draws on experience he gained as a union representative, and later, as a consultant for management. Some of the problems discussed are: jurisprudence and the labor contract, the prime union contract and its administration, "right to work" laws, management rights, wage incentives and seniority, the strike threat and grievance procedure, and arbitration. Excerpts of the Taft-Hartley Act and a model contract clause dealing with wage incentives have been included in Appendices.

The book is meant as a practical guide of day-to-day problems confronting a company, its employees, and trade unions. It is not a historical record of trade unions or a psychological study of the influence of an organization on its workers.

Technology in American Water Development. By Edward A. Ackerman and George O. G. Löf. The John Hopkins Press, Baltimore, Md., 1959. Cloth, $6 \times 9^1/_4$, 709 pages. $10.

The matching of water supply with demand, a complex undertaking as it is, has to contend with many, continuously varying factors: Technology changes, population increments and shifts, and political factors are perhaps the principal forces influencing water management.

In 1955, for example, about 10.5 billion gallons of water per day were withdrawn in the United States for municipal residential, commercial, and public use, with about a billion gallons more used by rural households. In industry, 400 tons of water are withdrawn for each automobile manufactured. A situation arising from modern defence operations is exemplified by the plight of Albuquerque, N. M., where between 1940 and 1955 the urban area grew from 40,000 to 200,000 people. Within the same period its water withdrawals from the Rio Grande Valley streams and ground water multiplied about seven times. The chemical industry, excluding all the "allied products," is the largest industrial user, consuming 8 billion gallons a day. The pulp and paper industry uses over 4.4 billion gallons a day with petroleum refining following closely behind.

Meanwhile with the demand for water ever increasing, 1060 trillion acre-feet of salt water are tantalizingly lapping

at our doorstep. Under the sponsorship of the U. S. Government Office of Saline Water, Dept. of the Interior, this immense potential is being unlocked. Many processes for desalting water have been investigated and those now being selected for development are expected to yield up to one million gallons per day of fresh water; enough to satisfy the needs of 20,000 people.

The book is one of the Resources for the Future studies; it reviews and discusses the physical and economic environment of water development, and techniques and technical events significant in the progress of water development. The four technical events chosen to demonstrate the rate of exploitation of water are (1) refrigerated transport and plant breeding; (2) growth of the chemical industry; (3) sprinkler irrigation; and (4) the residential demand for electricity.

The study also treats the emerging technology which is of potential impact on water use. The techniques affecting water demand are connected with developments in the generation of electric power from conventional and nuclear fuels, and the relation of waste disposal in the atomic energy industries to water requirements. In discussing the expansion of the physical range of water recovery, the techniques described and evaluated relate to the use of saline waters, weather modification (artificial rainmaking), and the discovery and estimation of underground water supplies.

Finally the work turns to an appraisal of the manner of studying the relation between technology and the administration of water development in the U. S., in particular, the place of administrative organization in matching supply and demand.

Package Design Engineering. By Kenneth Brown, John Wiley & Sons, Inc., New York, 1959. Cloth, 6 × $9^1/_4$, 263 pages. $8.50.

The book comprises material which the author originally prepared for a course he offered on "Industrial Package Design" at the University of California, L. A. The rudiments of mechanics, statics, and dynamics preface the treatment of materials handling and packaging. Structural analyses and design applications are treated throughout with the aid of elementary mathematics; where a knowledge of higher mathematics is required to understand the more advanced theories, e.g., the theories of vibration, only the simplified equations are given.

The containers discussed are fiberboard, plywood, and metal. Based on the specific application and on the degree of abuse that a container is expected to withstand (environment, dehumidification and pressurization, shock, corrosion, or fungus attack), a design is evolved which will allow it to have maximum protective capacity.

Both military and commercial applications are given as well as the specifications for containers to be used in each category. Cushioning, tension spring, canvas strapping, and rubber shear mounts suspension systems are compared. Many design problems, some of which have been encountered in practice, are fully worked out and similar ones have been left as an exercise for the reader.

Brimstone. By William Haynes. D. Van Nostrand Co., New York, 1959. Cloth, 6 × 9, 308 pages. $5.75.

Many readers of technical literature are acquainted with previous writings by the author Bill Haynes and know how he can deal with a complex subject by treating it in a popular vein without beclouding the information in any way. The present volume is based on the authors earlier work entitled "The Stone That Burns," written in 1942. Useful appendices are included.

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Third Mechanical Pulping Conference Papers

PRESENTED AT QUEBEC, QUE., SEPT. 10–12, 1958

A New Grinding Method

PIERO BERSANO

A Dutch papermaker speaking of the coming free European market made a very wise observation concerning "have" and "have not" nations in respect to raw materials. The "have" nations, like Austria and Scandinavia, will have no problems; but the "have nots," like Italy, France, Benelux, and Germany, have to learn to use what is available *now* if they hope to compete when the free European market comes into being. In our company in Italy we have started an extensive reforestation program including studies of the fastest growing softwoods best suited for our purpose; but we cannot afford to wait 20 or 30 years if we hope to be competitive in this market.

The new type machine which I am talking about today was conceived as we were seeking a method of advantageously utilizing our existing raw materials.

The basic assumption on which we worked at the time was that any fiber has good characteristics for paper production, provided it has been subjected to the right chemical and mechanical treatment; just as any man, regardless of his size or color, can be trained to do any physical job.

Our first thoughts were directed to the semichemical field which, of course, imposed problems, impregnation—in itself difficult—and cooking with the resultant loss of yield and the high cost of chemicals and steam.

During our many tests we tried and used smaller chips endeavoring to get them smaller and smaller. From this came the idea of substituting a grinding wheel for the chipper (see Fig. 1).

In our first efforts we simply substituted a grinding wheel for the chipper knives and placed a log against a wheel, as in chipping with the hope that the log would be pulled into the wheel as it was ground away.

This was not successful and after trying various angles of the log with respect to the wheel, we came to the conclusion that self-feeding of the machine was impossible to achieve. We discovered that if the log was placed parallel to the wheel and it was short enough in comparison to the wheel diameter, it rotated and some grinding action took place. We built a small machine utilizing this principle and used it for some time in our laboratory.

This naturally led to the next step which I say is the fundamental one. It started our thinking toward rotational grinding. Having in mind the centerless grinder used in the mechanical industry, we built and experimented with a machine based somewhat on that principle (see Fig. 2). It worked and worked well but had some fundamental drawbacks; it could not be fed continuously and, in addition, it was practically impossible to completely grind a log because the grinding wheels did not permit grinding below a given diameter of the log.

Struggling with these difficulties led to another idea; we substituted the cylindrical grinding wheel which was as long as the log, with a series of narrow cylindrical grinding wheels working at an angle to the log. These wheels supplied the same rotational and longitudinal movement of the log (see Fig. 3). The series of wheels then could be arranged at different elevations to compensate for the wearing away of the log. A small laboratory unit was made to test this idea and later a commercial model properly designed, was built which worked well. In order to have smooth operation in a commercial machine, we had to supply part of the rotating and longitudinal forces by applying power to the carrier rolls, unnecessarily complicating the machine.

The next step then, of course, was to avoid the necessity of

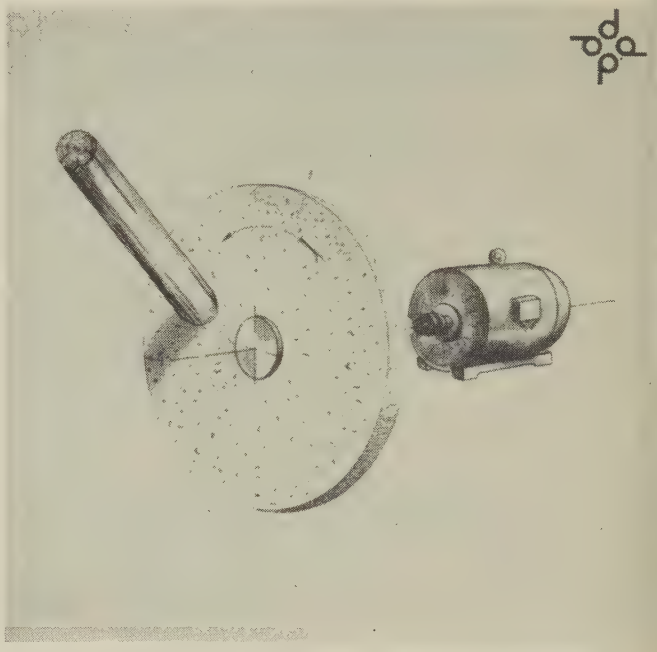

Fig. 1

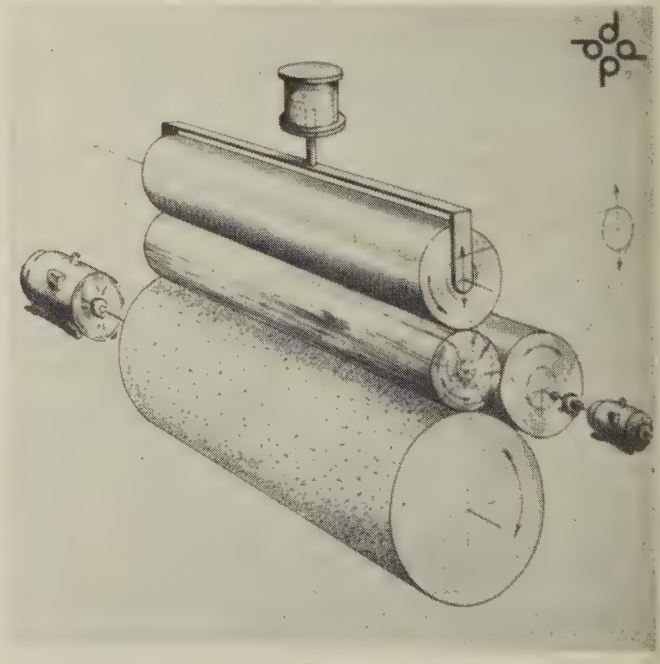

Fig. 2

Piero Bersano, Cartiere Burgo, Torino Italy.

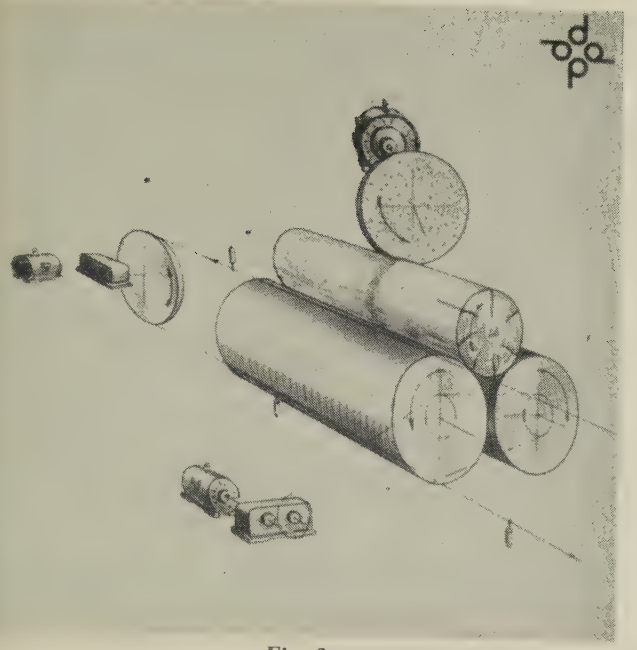

Fig. 3

supplying the external power for feeding and rotating. This could be accomplished, we decided, by an increase in the contact area of the wheels on the log by using hyperboloidal wheels (see Fig. 4). The commercial machine was modified accordingly and results were improved. However, we felt that it could be even better and we experimented further with shapes of grinding wheels finally arriving at the present conical type.

Our objective, as we stated, was that the wheel should supply rotational and longitudinal movement to the log, allowing at the same time the free discharge of the pulp. The conical wheel satisfied all of these requirements and was proved to our satisfaction in the laboratory unit.

Encouraged by our successful efforts, we were eager to start the construction of a commercially practical unit. Our thoughts then, were of a machine allowing the log to be fed vertically rather than horizontally, as had been the case heretofore; the wheels arranged vertically would facilitate the feeding of the logs and make a more practical device.

We proceeded with the manufacture of this machine and then conducted a series of experiments which pointed out some further difficulties. The last wheel in the series either had too much or too little pull resulting from the irregular work of the preceding wheels. We have returned for the moment to the horizontal type and have built a new machine of commercial size (see Fig. 5).

Observations made during the hundreds of tests in the various stages of development led us, I believe, to a better understanding of what happens during the grinding action.

We learned, for example, that the grinding wheels must be kept scrupulously clean and showers must be most effective. Consistency of the pulp produced can be regulated at will providing the grinding wheel is clean, as mentioned. We have worked with consistencies as high as 12% without any trouble. This last is very important if the temperature is to be raised by some external means.

Second, but no less important, are the right angles of contact between the wheel and the log. The present machine incorporates the correct angles of wheel to the log and angle of the cone on the wheel to the axis of the log.

This system must have enough inertia to maintain the rotational speed and longitudinal movement of the log notwithstanding the knots or soft spots in the log. This can be achieved by the correct calculation of the weight, diameter, and position of the supporting or carrier rolls.

At this stage we believe that the rotational speed of the log must be constant and this can be accomplished by variations in pressure. This does not have to be constant for all woods but can vary, depending on its hardness. The feeding and, consequently, the output varies due to the constant rotational speed. The variations are at a minimum for dense wood and maximum for softer woods.

The reasons for that will become evident when we visualize how the grinder operates (see Fig. 6). Let us examine the log rotating under the wheel. In the first stage of contact, the wood is compressed by a motion tending to increase the peripheral speed of the log. As the log progresses, it is still subjected to compression and the motion of the wheel tends to push it in an axial direction. The difference in the rotational speed of the log results in a grinding action in the layer of compressed wood in the grinding zone. The fibers are pulled away from the log at some angle to the axis. The grinding rate is governed by the feeding speed. At each revolution, the log is subjected to alternating compression and decompression in contacting the wheel and each of the supporting rolls. This, by itself, tends to loosen the bonds between fibers thus preventing breakage when finally pulled away. If the above conditions are maintained, the power consumption is extremely low approaching the theoretical minimum, as shown by recent tests in the United States.

I believe that, at this point, it is clear to everybody that the machine has great flexibility. By varying the angles, the pressure, and the abrasive specification, it is possible to produce a wide range of fiber classifications.

The speed of the log can be controlled by varying the angles and the pressures of the grinding wheels. The abrasive specification can be varied to produce the correct type of pulp in a normal range, as is done with pulpstones. Or it can be very coarse to produce bundles of fibers suitable for further treatment either with chemicals or simply mechanical refining, or both.

It is difficult at the present stage of our research to state which is better—producing primary pulp by grinding or by grinding and refining—the results being influenced in the latter case by the degree of action of the refiner, that is if it produces bundles only, or if some breaking action transpires. We observed in the case of poplar that when the thin wall of the fibers have been soaked in water, many times, they could not withstand the action of the refiner and were broken.

The groundwood obtained by our experimental machine,

Fig. 4

when it was working properly, had good opacity, and burst and tear factors comparable but not lower than our standard poplar groundwood. We have conducted most of our work so far with poplar in order to correlate results.

The pulp from our machine is generally cleaner than standard and we believe that this stems from the fact that the knots are ground to a powder and are completely dispersed throughout the main body of the pulp, and because they are very fine most of them may be washed away.

Another important factor that we have found affecting the pulp produced is the temperature. Increasing the temperature of the shower water always gave excellent results in respect to the characteristics of the pulp produced. Whether this is a result of the weakening of the bonds under heat or from the lower solubility of air in the water, and consequently less oxidation on the fibers, remains to be seen.

We are presently conducting tests to clear up this important point as it can be of paramount importance. The main differences between our machine and a conventional grinder is that we do not have the heat effect from friction and hence obtain a substantial power saving as a consequence. But at the same time there is a strong oxidation on fibers for many reasons, such as solubility of air in cold water, ventilating effect of the wheel, all of which can be controlled, creating in the shower water and in the atmosphere surrounding the wheel ideal conditions for best results. Because of the different conditions mentioned, we hope and expect to produce a superior type of groundwood which, of course, is our objective.

As mentioned before, the wood is subjected to compressions and decompressions before and during grinding similar to what happens in a Kollergang, which we have used for many years in a production of cold soda pulps. We believe that our machine presents an ideal situation for impregnating before grinding as we are interested in impregnating only the wood that will be removed by the next grinding wheel. By this method it appears possible to treat any wood, even those that by standard methods cannot be impregnated. We have in effect been able to produce cold soda pulps. Using different chemicals in the grinding water, the soaked bundles of fibers produced by grinding can be easily cooked, resulting in a strong semichemical pulp which can be readily refined.

We have made hundreds of tests under different conditions and on different woods and, while we cannot be absolutely positive, we can foresee the possibility of obtaining prime quality pulps from a very large range of hardwoods. This

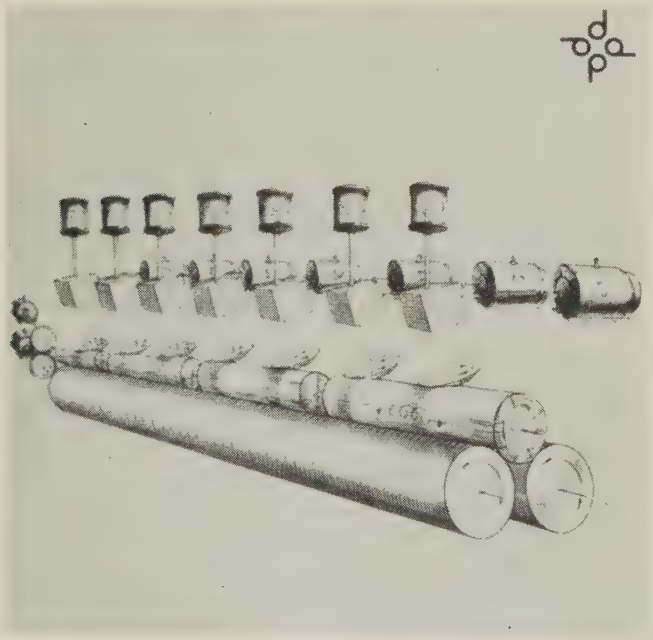

Fig. 5

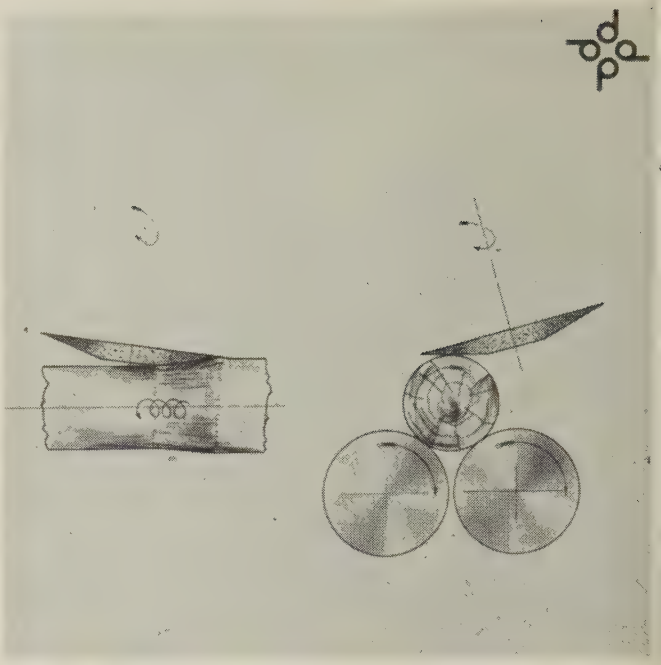

Fig. 6

was exactly what we hoped to achieve when the program was initiated.

We still have a long way to go in developing the machine itself, but the results so far are so promising that we are confident that we will soon reach the point where we can have a successful commercial machine.

I want to point out that the laboratory unit has been working satisfactorily for many months and it has proven to be a very valuable tool for research work. The advantage of the low power required (motor 30 hp.), the possibility of controlling or separating the pulp obtained from the external and internal rings of the log make it a very fine tool for laboratory groundwood research. For the time being, this is of prime importance.

This program is jointly sponsored by my company, Cartiere Burgo, Norton Behr-Manning, and Norton Co., and I wish to recognize and express my sincere gratitude for the enthusiastic and able support and assistance of the staff of the Central Laboratory of Cartiere Burgo in Turin.

The legal and technical assistance of the Norton Behr-Manning and Norton Co. staffs and the encouragement of the officials of these companies are most gratefully acknowledged.

CHAIRMAN: There should be some questions on this very, very interesting paper.

G. E. HAEGER (Fridafors Fabrieks, AB, Sweden): You referred to energy consumption as approaching some theoretical values. Could you give us any figures, either of the theoretical values or actual energy consumption as kilowatt-hours per ton or horsepower per ton per day.

MR. BERSANO: From what we have seen on the laboratory machine we believe that we are in the range of 250 to 300 kw.-hr. per metric ton. But maybe it is less because as soon as the machine begins to work irregularly (that is what happens in the laboratory unit at the beginning and the end, because at the beginning the logs tend to rise and at the end it tends to rise the other way), the power goes up, but when it is running smoothly the power is very, very low and, in any case, the figure I gave that we have controlled, can be the right power at the present time. And, I believe, maybe, we can go lower.

H. WARBURTON (Price Bros.): I want to congratulate Mr. Bersano on an excellent paper. I have just a couple of questions. Was there any pattern on the wheels? How small a diameter can you get your logs down to?

MR. BERSANO: Well, there is no pattern on the wheels, and diameter of the log is reduced to zero because we go down with conical wheels to the diameter of about 1 in., and then by means of cylindrical wheels we have developed as second step, we are able to reduce completely to zero.

MR. MCCULLOCH: Mr. Bersano is certainly to be congratulated. The paper was something refreshing. It was something new. The question I would like to ask is that there seemed to be a great uniformity in the size of the logs fed to the machine.

Has Mr. Bersano solved the problem of handling various sized logs?

Mr. Bersano: We don't need to have uniformity, because the only thing required is that the logs must be more or less straight. If there are crooked logs they must be cut so that you have only more or less straight fractions. Otherwise, the diameter of the log is not influenced. We have a feeding device before the machine that force-feeds the logs. If a log is small it will pass through the first, second, and third wheel without any grinding action, it will then find a wheel that begins to grind.

Mr. McCulloch: In the case of our own mill we get a wood supply varying from 4 up to 18 in., which would present, I would think, quite a problem, unless you have some kind of a take-up on your wheel.

Mr. Bersano: Well, it means making the carrier rolls larger to carry the larger logs. I don't foresee any difficulty in doing that. As each section takes away a certain volume of wood, more sections would also handle a large variation in sizes.

Mr. Curtiss: Mr. Bersano, is there any considerable difference between the strength characteristics of pulp produced from this technique as compared with conventional grindstones?

Mr. Bersano: No, there is no difference. Of course, you must find the right angle of the wheels and the right grit of the stone, but you can obtain a conventional groundwood much better than with the conventional grindstone, provided you have the right conditions.

W. D. May (Pulp & Paper Research Institute): Mr. Bersano, I have two questions. Do you first of all have to condition the stones in any way before they prduce your best optimum pulp? And the second question: I noticed you stressed that the stone has to be scrupulously clean. What goes wrong with the pulp if the wheel isn't clean?

Mr. Bersano: The grindstone, as I said, has no burr; there is just a grinding stone as is normally produced. On conventional equipment the burr provides the discharge of the stock. On our grinder the stock is discharged by centrifugal force and what makes a difference in operation is having the stone clean or not. We have seen that if we close the spray water as soon as the wheel is not clean, then the power consumption goes up and the wood starts to burn, and that may be what is happening sometimes on conventional grinders. When the stone gets dirty it consumes power and produces bad pulp.

G. E. Soyka (Bolton-Emerson): Mr. Bersano, have you any information on the effect of the speed of the feed and the rotation of the grinding disks? Is it possible that you get different qualities of groundwood from the same wood by the difference in the feed at different settings of speed and at different speeds of the grinding disks?

Mr. Bersano: That is right. You can vary the speeds, rotating and longitudinal, and produce different pulp.

R. O'Donohue: I would like to ask Mr. Bersano about the method of using chemicals in your grinder. I think that it might be a very important consideration, inasmuch as it would give possibilities of getting different qualities of pulp—perhaps stronger fibered pulps. The other angle is that he mentioned something about using a bleaching solution in his grinders, and I would like to know just what the effect of that was.

Mr. Bersano: We are planning to install on the commercial machine, a vat full of water enclosing the carrier rolls. The wood is compressed by the carrying rolls and when it leaves them it decompresses and absorbs the liquid in the vat. Working on that principle we used soda solution in the vat at different concentrations and obtained (rotation, longitudinal speed, and grit being constant) pulps of different characteristics as it happens in the cold soda process using chips. What we have tried as regards to bleaching, is to have some preparation for bleaching. We are making studies now. We are not at all sure, as I just mentioned, but it seems if we have a preparation for bleaching we can bleach very easily afterwards. As a matter of fact, and I give you this without any guarantee because we have made only some tests, we have seen that we can bleach and gain 18 to 20 points with peroxide at room temperature, without heating at all.

Mr. Maung: I want to ask a question on the amount of production. How do you control the production, say, tons per day, and what is the anticipated production?

Mr. Bersano: The production is controlled by the longitudinal speed by which the logs travel through the grinder. These machines are not completed. We are sure that we can make about 12 metric tons per day, but that is only the first stage, because we don't know exactly how much we can grind, how deep we can go into the wood, and how fast we can put the log through the machine. That is what is controlling production.

Mr. Loly (Bowaters): I would like to ask you, do you think that your machine will possibly be limited due to the immense length you have to have in order to get one log through? In other words, you are either going to have to have gangs to get your logs down, or a conveyor system taking them back to the beginning of the process. Do you think you are going to be limited by literally having to have a machine about the length of a modern high-speed new machine?

Mr. Bersano: That is what we ought to have!

D. Atack: I wish to ask if you have any color problems when you run with a clean stone. Further, do you have any slides of your poplar groundwood which could be projected at this meeting?

Mr. Bersano: No, I haven't. I am sorry. If you want I can send them to you later. About the color. The color is influenced by the wood and, to some degree, by what you have in the water. If the water is acid you have a brighter color, and if alkaline the color goes down. In normal grinding there is not too much difference, for the reason that we are grinding, at the present time, at a rather low temperature in the conventional grinders. We have used freshly cut poplar. We are high in brightness. We can be from 63 to 66. If it is old wood that was stored for 6 mo. or 1 yr., we are in the range of 58.

C. Giogio (Cartier Italiene): I would ask Mr. Bersano if they actually tried to classify the fibers coming out from different sections of their machine? You said that you can have different kinds of pulp depending on the diameter of the log.

Mr. Bersano: We have made hundreds of fiber classifications. I can not answer your question because with the machine you have seen, I had the three sections of the commercial machine operating just a week before I left, so I don't have the complete situation as it is. Anyway, from the tests we have made on the small grinders we have seen that the small fraction, the fraction passing under the 200-mesh screen is lower than the conventional grinders. But that depends on the relative condition of wheels and speeds and so on, because we can produce, too, much worse pulp than the commercial grinders.

Mr. Giorgio: I think it would be really interesting to use your kind of machine in order to classify pulp, and to have different kinds of pulps coming down from the different sections of the machine. Don't you think so?

Mr. Bersano: We will do that. As soon as the machine is operating we shall do that just to see what happens, because it is very interesting to see what happens if we divide the pulp obtained from the external rings and the pulp obtained from the internal rings of the wood.

H. W. H. Jones (Consolidated): I would like to ask Mr. Bersano if he has had any experience with spruce and, if so, whether the pulp quality is the same as with conventional grinders.

Mr. Bersano: Well, as I said, we decided to experiment only on poplar to have comparable results. We made some tests on spruce, too, for spruce is just the same thing. It is only to find the correct position and you can have good pulp out of spruce.

O. Forgacs (Pulp and Paper Research Institute): Mr. Bersano, what is the approximate area and shape of the grinding zone between the stone and the log?

Mr. Bersano: I should say it has an elongated shape with the main axis being the theoretical contact line between log and stone. That is the compression area that makes the rotation and the log move.

Mr. McCulloch: Mr. Bersano, we all know that in conventional grinders they have their ups and downs quality-wise. Do you think your machine is inherently better for maintaining a constant quality, than an ordinary grinder?

Mr. Bersano: I suppose so, because we are investigating the matter, but as I mentioned, in order to obtain a constant quality, maybe the best thing is to try to keep the rotational speed constant and, in that case, the longitudinal speed will vary. In other words, if a knot or a harder zone of the log comes into contact with the wheel, if the rotational speed is kept constant, the log gives more resistance to grinding, so the longitudinal speed decreases but, in that case, what happens is that the log rotates many times more before passing under the wheel and so it is subjected to the compressing action much more and, in that case, we feel pretty sure that we can obtain a pulp that can be more or less constant. I mention what happens to knots. The knot, when ground, is reduced to perfectly good fibers that are darker in color and black material that is reduced to a powder form by the action of the wheel.

Mr. Forgacs: You feel, then, that the alternate compression and decompression of the wood softens and loosens the fibers sufficiently for the grinding wheel to carry out a more final separation?

Mr. Bersano: Yes, that's right. When we made the vertical machine I mentioned, the wood at the end, when it was about 2 or 3 in. in diam. was so softened that many times it broke. At that stage you could have separated the fiber with your fingers.

R. T. Wetmore (Ontario Paper Co.): Mr. Bersano, you talked about the need of keeping your stone very, very clean with the showers. In your experimental work so far have you tried using recirculated white water for cleaning the stone, or do you find that you have to use fresh water to keep that degree of cleanliness?

Mr. Bersano: We did use recirculated water. As a matter of fact, when we are working with the vat and carrier rolls immersed

in water, we use recirculated water, and that is easy, because you don't mind the amount of water you are putting on the grinding wheel.

Mr. O'Donoghue: Before, Mr. Bersano, you mentioned about the pressure applied to the wood and that it is localized in certain positions there. How does the amount compare with the conventional grinders?. Is the pressure applied measured per square inch or is it by some other standard?

Mr. Bersano: Well, that is a very difficult question, because the pressure per square inch depends upon which area is really contacted with the wood, because if we had, instead of a log, a bar of iron we should have a straight line, so the right answer would be a linear pressure. In the case of wood it is diffuilt to say. We are trying to get some exact figures of what happens but, at the present moment, I cannot give you any figures because we are working, more or less, trying to keep the same conditions for all wood. That is, not to have too much pressure, otherwise the wood is crushed, and not to have too little pressure because, otherwise, the wood doesn't rotate and move. But we are investigating the matter to see what is the ideal compression of the wood in order to get an area that is ideal for grinding.

Mr. Atack: From your remarks I assume that it is possible to reduce the volume of shower water until the pulp emerging from the stone has a consistency of 12%. Do you burn the wood if the shower water is reduced still further?

Mr. Bersano: The burning action takes place any time the wheel is not clean. I can tell you what we have made for some minutes, we have worked at a much higher consistency, in the range of 30 to 40%, because we tried to clean the wheel with steam instead of using water, but after a few minutes the wheel broke. But we have seen that as long as the wheel is clean nothing happens in regard to burning of the wood.

R. M. Drummond (International Paper Co.): I would like to ask two questions. One is concerning the percentage of rejects. Photographs showed, in my opinion, that the ends of the logs came out rather broomed, and secondly, you mentioned stone grit varying from 16 to 60. I wondered, with this process, if the horsepower consumption, due to differing the stone grit, was more or less in the same proportion as you would get with the normal grinding process? Or is it less?

Mr. Bersano: I can tell you that, so far, we haven't noticed any difference in power consumption, either using the 60 grit which is producing an acceptable pulp like conventional grinders, or using the 16 and making a very coarse groundwood that can be very easily refined afterwards in the refiner. We have not noticed any difference in power. As I said, our figures are not exactly sure because we are working with a small machine.

Mr. Drummond: What about your rejects?

Mr. Bersano: It depends on the grit you have. As I said, using the 60 the rejects are just in the range of conventional types.

Mr. Drummond: Yes, but with the same quality of groundwood with a normal process, higher or lower rejects?

Mr. Bersano: Yes, having different grits you can go up to 100% rejects, but the power consumption does vary.

Prof. Brecht: I would like to congratulate Mr. Bersano on the excellent job he has done in developing this new machine, and to ask one specific question. It would appear to me that the stone you are using will change in surface during production just as occurs with an ordinary standard grindstone. In this event you would presumably require sharpening, and I wonder what provision is made for this.

Mr. Bersano: Well, so far we did not see any necessity for resharpening. Of course, we are not in the stage of running 24 hr. a day for days and days and days, and that is one thing we want to see in the machine we are building. But I presume that we shall not have to do any sharpening because you don't do ordinary sharpening on grinding wheel working on metals. Here is exactly the same thing. If the wheel is clean it works always.

Prof. Brecht: It does seem to me, however, that if the stone has essentially similar composition to the customary stones, some grit must be lost during use, and that consequently resharpening would be necessary regardless of whether the stone is kept clean. This would be particularly required in your system, since it is my understanding that you produced long-fibered stock. The angle at which you are carrying out your work results in a more nearly parallel action than in normal grinding. You are doing an excellent job with poplar, but it is difficult to believe that it is possible to decrease the specific power consumption at the same time.

Mr. Bersano: Well, I don't want to state it bluntly, but I believe that you are wrong. Anyway, in my opinion there is a big difference between a grinding stone mounted on a conventional grinder and this kind of wheel, because in this conical wheel, and it was my purpose to have a conical wheel, the discharge of the pulp is immediate; immediately it is discharged from the wheel by the centrifugal force. In the conventional stone I believe the burring has another important function. It is to provide the discharge of the pulp. That is the reason why I don't believe that shall need any dressing of the stone.

J. H. Perry. The pulpstone absorbs fines when the wood is pushed against it (absorb is not quite the right term), fines and pitch are impressed into the texure of the stone, finally filling the pores. This condition is indicated by a dull acting stone and can only be corrected by sharpening. In the case of Mr. Bersano's machine, that is not so, because there is no grinding pressure approaching that of conventional grinding, therefore there are no fines or pitch embedded in the stone. Because of this, very little or no sharpening is required. As Mr. Bersano pointed out, centrifugal force tends to keep the grinding wheel surface clean, and due to the fact that the grinding wheel does erode to a degree in water, it tends to be self-sharpening, which is not true of conventional pulpstones.

Chairman: The time is really going along pretty fast and in order to get this other paper in I think we should stop this and look for somebody who has some very important question they want to ask in the short period of time.

Our next paper is by Mr. Beath of Price Brothers on the effect of angle on freeness of pulp and here again we have a paper dealing with a new aspect, at least an aspect that not many of us are familiar with, and at this time it gives me great pleasure to introduce Mr. Beath.

A Cause of Wide, Uncontrolled Freeness Variation in Groundwood Production—The Varying Angle Between Stone Surface and Wood

L. R. BEATH

The existence of uncontrolled freeness variations in grinder product has long been recognized, but their wide scope under some circumstances is not generally appreciated. Results of an investigation into such variations in continuous grinders are given. Freenesses ranging from 40 to 250 were found in the product from a single stone over a 6-hr. period during which no deliberate change of grinder operating conditions was made. The major cause of these large variations was found to be rather small changes in the angle between the axes of the logs ground and the surface of the stone. It was found that a 10° decrease in angle might cause a sixfold increase in freeness. This angle effect has important implications in the commercial application and the fundamental theories of groundwood production.

It is well known that the quality of groundwood from a given stone will vary from time to time independently of the normal grinder control factors and of the normal effect of the stone dulling cycle. The large magnitude of these variations, for some stones, is not generally appreciated and first came to the writer's attention in data kindly supplied by Messrs. Wiklund and Lund of the Woikka Mill of Kymmene Aktiebolag, Finland.

Investigation showed that large, apparently spontaneous freeness changes were occurring in the pits of the continuous (Caterpillar) type grinders of the Kenogami Mill. These variations were found to have their major source in small changes of the relative position of the wood and the stone surface.

Prior work on the effect of wood-stone orientation includes: that of de Montmorency (*1*) on longitudinal grinding in which wood is initially tangential to the stone surface with its grain lying in the direction of stone surface travel. Brecht (*2*) reports on the effect of angles intermediate between the normal transverse and the longitudinal grinding conditions. Daniell (*3*), referring to continuous magazine grinders, states: that if pulpwood is not parallel to the axis of the stone the fibers are ground on the ends; the result is that instead of long fibers being separated there are many small pieces of fiber broken off the ends of the fibers; much power is wasted and the pulp is of poor quality. It is standard operator's

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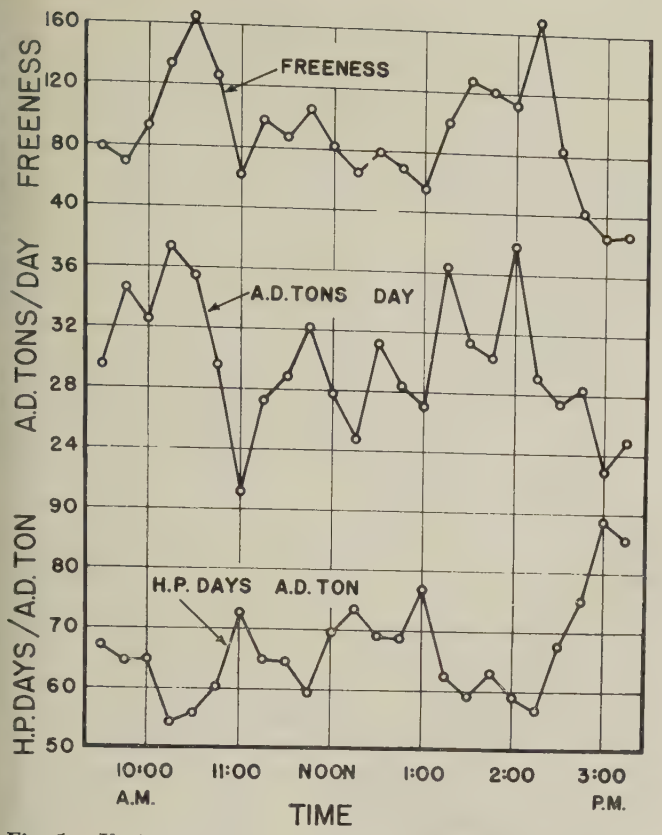

Fig. 1. Variation of pit freeness, grinding rate, and unit power input in normal operation of a continuous grinder

knowledge that if wood is criss-crossed or strongly angled in the magazine of continuous grinders, it may wedge against the magazine walls: rate of wood advance and effective grinding are reduced, and production rate and freeness decrease strongly; in the extreme, grinding may stop. It is apparently to these fairly extreme aberrations that Daniell has reference. The present paper, however, is not concerned with variations in horizontal angles but has to do with commonly occurring, small variations in the vertical angles. There appears to be no prior reference to the large effect on groundwood quality of these variations of rather small vertical wood angles.

GRINDER TYPES TESTED AND SAMPLING PROCEDURE

Most of the tests on freeness variation, and all those involving wood angle measurements, were made on Waterous continuous grinders, magazine width 50 in., grinding wood of 49-in. length. Feed chain drive was by oil turbine regulated by manual throttling. Most testing was done on a single stone driven by a 3500-hp. motor. Tonnages were calculated from thermal and material balances using power input, and the pit and white water consistencies and temperatures. Some testing was also done on Waterous hydraulic grinders to evaluate their relative uniformity of grinding.

The samples taken comprised several collections across the full width of the grinder dam: such samples were taken at 5-min. intervals and combined into composites representing 15-min. periods: freeness tests were done on these. Testing at shorter sampling intervals had shown no significant variations not adequately represented by the 15-min. composite samples.

FREENESS VARIATIONS DURING NORMAL OPERATION

A considerable number of tests were made to determine freeness variations from our continuous grinders. Figure 1 shows a representative record over a period of 6 hr. for freeness, production rate, and unit horsepower input. Normal grinder operating routine was followed throughout this period. At time of testing, the stone surface was rated as average, neither sharp nor dull.

Table I. Maximum, Minimum, and Average Freeness and Standard Deviation of Grinder Test

Test Number	Freeness Max.	Freeness Min.	Freeness Av.	Standard Deviation, %[a]
1	204	30	98	46
2	196	22	97	52
3	203	77	144	23
4	204	89	136	19
5	197	118	156	18
6	82	24	55	33
7	168	26	91	41
8	71	9	40	41
9	111	21	55	48
10	101	23	65	38
11	118	22	67	47
12	292	76	141	43
13	192	61	103	41
14	101	29	60	33
				Average 37.4

[a] Expressed as a percentage of average freeness.

To compare the freeness variations in different groups of samples, some convenient measure of variation is needed. The standard deviation of sample freeness is such a measure. For data groups of low average freeness, the freeness variations and standard deviations tend to be low: for high average freenesses, variations and standard deviations tend to be high. However, due to the nature of the freeness test, a small variation at low average freeness may reflect as large a quality change as does a large variation at high average freeness. A change from 40 to 60 freeness has an importance much closer to that of a change from 200 to 300 than to one from 200 to 220. For these reasons standard deviations have been expressed as percentages of the average freeness of the set of samples concerned. It has been found that standard deviations so expressed tend to be independent of the mean freeness level.

Table I gives data from a number of tests taken over a period of 3 mo. The samples tested represented successive 15-min. intervals over periods of 5 to 6 hr. in each case Tests no. 1 to 12 were all made on the same continuous grinder: tests no. 13 and 14 were from other grinders of the same type. These tests were all taken during periods of normal, routine operation of the grinders.

The mean standard deviation for this group is 37% of average freeness. This 37% figure means that, for a total of 8 hr. per day, the freeness from a representative stone may be expected to be at least 37% above or below the daily average and that, for over an hour a day it will be at least 74% above or below average.

These are very large freeness ranges. Statistical estimates of their effect on the freeness of the blend from a fairly large

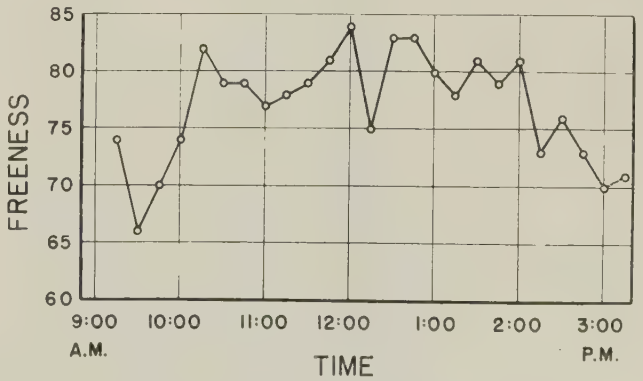

Fig. 2. Freeness variation of regulator groundwood at paper mill mixer, normal operation

number of stones so varying indicate that residual variation in the blend may still be important in terms of use requirements. Direct tests on the screened, deckered groundwood, as furnished the paper machines gave standard deviations of 5 to 7% of average freeness, this again for composites representing 15-min. periods. With an average freeness of 85 and a standard deviation of 6%, freeness was below 80 or above 90 for 8 hr. per day and below 75 or above 95 for over an hour per day. Such variations can be expected to disturb paper machine operation. A typical record of freeness variation of stock at the groundwood regulator is given in Fig. 2.

EFFECT OF NORMAL GRINDER CONTROLS

The usual grinder control variables were carefully checked as possible causes of the freeness irregularities. Grinding was done through several shifts at constant feed chain speed by second-by-second manual control of the feed chain oil turbine. By the same means a constant motor load was kept on the stone through other shifts. No major increase in uniformity of freeness resulted from either mode of operation. Freeness variation seemed equally bad with both sharp stones and dull stones. Grinding temperature was not a factor, since it was automatically held to a set point at all times. Pit consistency varied inversely with freeness, but as this was simply a result of the automatic temperature control adjusting white water flow to the varying power inputs and grinding rates at which the pulp *had* already been made, the consistency variations were rated as effects rather than as possible causes.

As none of the grinder operating controls seemed responsible for the freeness changes, and as appropriate short term changes in the stone surface seemed unlikely, attention was directed to the wood condition in the magazine. Among the variables which might affect freeness were considered: variations in the void volume among the logs in the magazine, and the angular positioning of the logs relative to the stone.

The grinders have a manhole 31 in. wide by 15 in. high located with its midpoint about 15 in. above the stone surface. This may be opened to show the ends of some of the logs passing down the magazine. In several tests the wood passing the manhole was assessed for:

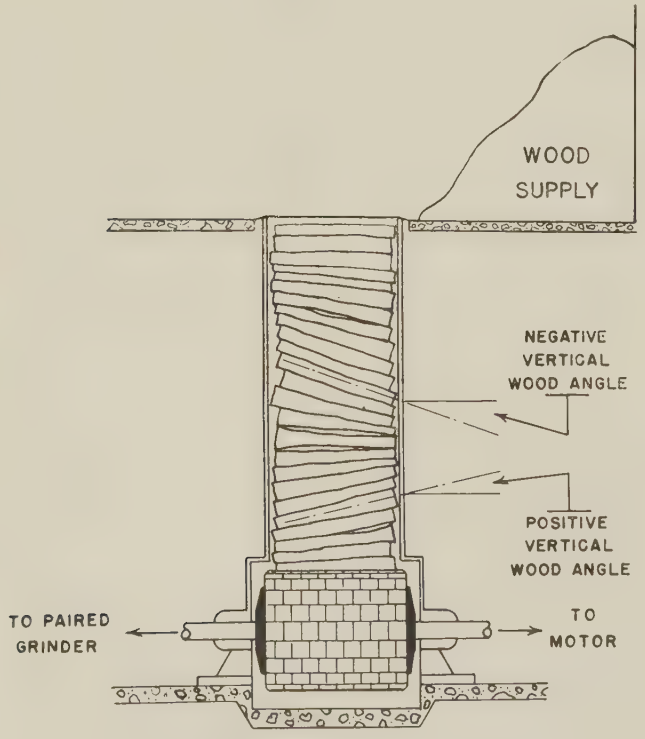

Fig. 3. Cross section of continuous wood grinder showing vertical wood angle

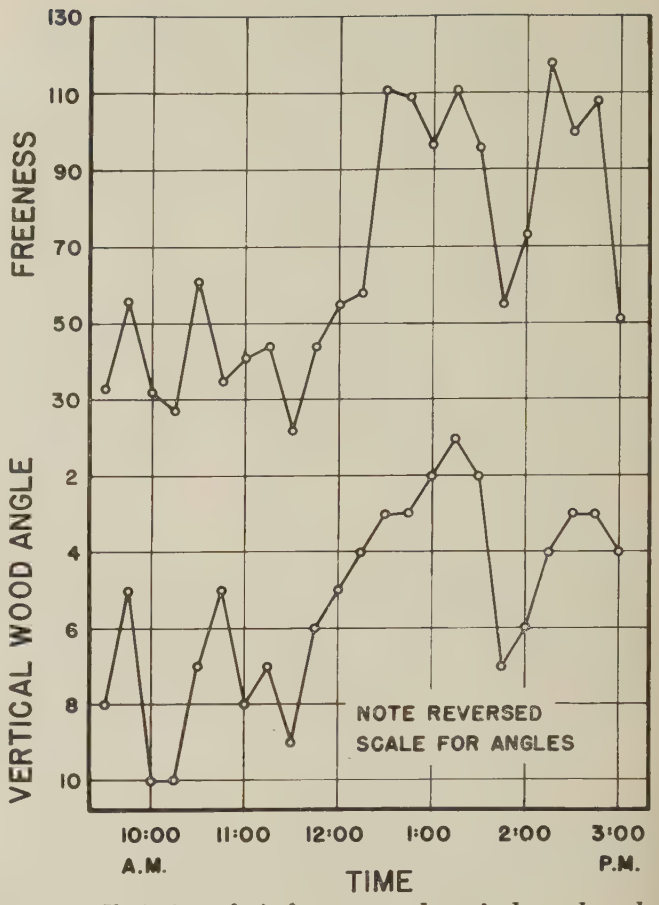

Fig. 4. Variation of pit freeness and vertical wood angle in normal operation of a continuous grinder

1. Void fraction. By visual estimate of the areas of the spaces among the ends of the logs.
2. Horizontal angle of the logs. This was measured by thrusting a straight, squared stick into the void space along the side of a log, and measuring the angle between the stick and a perpendicular plane parallel to the grinder shaft.
3. Vertical angle of the logs. Similarly measured by placing the stick parallel to the log axis and noting the angle of the stick to the horizontal. The vertical angle is clearly shown in Fig. 3.

Large variations were found in all three factors. Multiple correlation analysis showed that there was no connection between pulp freeness and the void fraction, nor between freeness and the horizontal wood angle. A very significant degree of correlation was found between the vertical wood angle and the pulp freeness.

THE EFFECT OF VERTICAL WOOD ANGLE ON FREENESS

Figure 4 presents freeness tests on a series of composite grinder pit samples, each representing a 15-min. interval. Also plotted are the averages of the vertical angles of those logs accessible for angle measurement. The grinder under test was loaded and operated in routine fashion during the test period.

It will be apparent that all major changes in the angle of the wood are reflected in changes in freeness.

Data from another test, also taken during the normal grinder operation, are shown in Fig. 5. Again, a strong similarity in the general courses of the freeness and angle plots is apparent.

In Figures 4 and 5, and others, the scale of angle has been reversed from the usual position to better show the angle-freeness concordance.

The data of Figs. 4 and 5 were obtained during normal grinder operation while normal wood loading practices

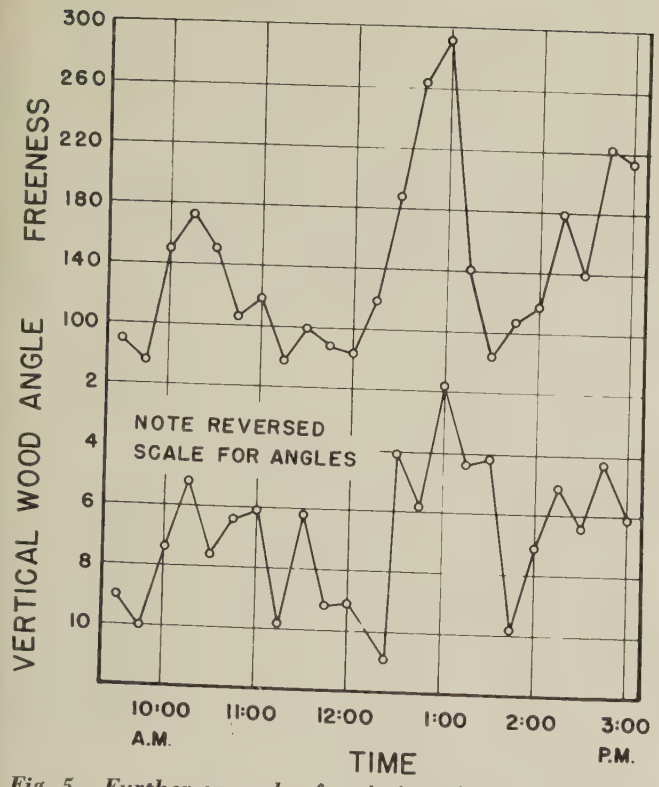

Fig. 5. Further example of variation of pit freeness and vertical wood angle in normal operation of a continuous grinder

obtained. Figure 6 presents test results during a period of controlled loading. Again the freenesses of composite samples representing 15-min. periods are given, together with the average vertical angle of the wood ground in the corresponding intervals. Through the period supplying the wood ground from 10:00 a.m. to 12:15 p.m., the logs were placed in the magazine as nearly as possible horizontally. Small departures from the horizontal at loading, plus shifting of the wood in transit down the magazine, has resulted in a small but definite average angle of the wood just above the stone surface. Following this period, the wood was deliberately loaded poorly to build up and to vary the vertical angle over the range often found as a result of routine loading.

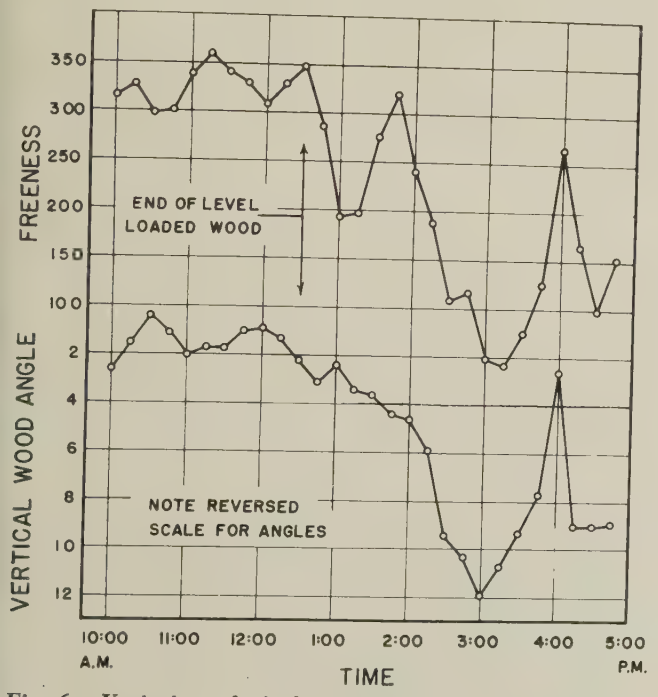

Fig. 6. Variation of pit freeness and vertical angle in a continuous grinder with wood loaded at controlled angles

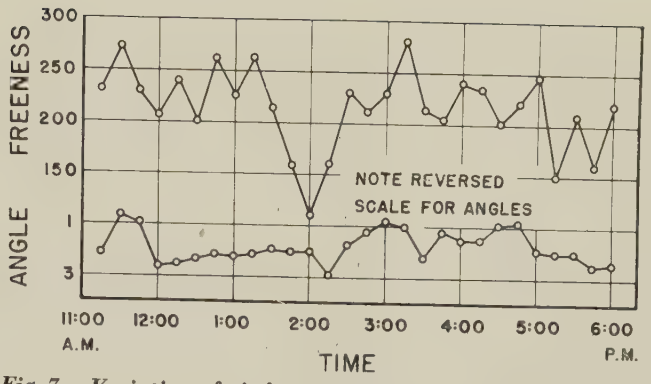

Fig. 7. Variation of pit freeness and vertical wood angle in a continuous grinder with wood loaded as level as possible

The high and relatively uniform freeness of the first period, averaging over 320, is in sharp contrast to the varying freeness of the second period in which a low of 42 was reached. During the whole of the test no change was made to any grinder control other than the changes in wood loading. The parallel variation of wood angle and freeness is particularly apparent here.

The freeness and vertical angle data of a further test in controlled loading is given in Fig. 7. Throughout this test every effort was made to have all logs loaded as close as possible to the horizontal position. Apart from the five samples of 1:45, 2:00, 2:15, 5:15, and 5:30 p.m., the freenesses maintained a high and quite reasonably uniform level. No reason was apparent for the low freenesses at the noted times.

The consistent, near-horizontal, loading gave the expected result of high and much more uniform freeness. The standard deviation of the freenesses here was 16.6% of average, as compared to the average of 37% found for grinders operated with uncontrolled loading. If the aberrant freenesses of the

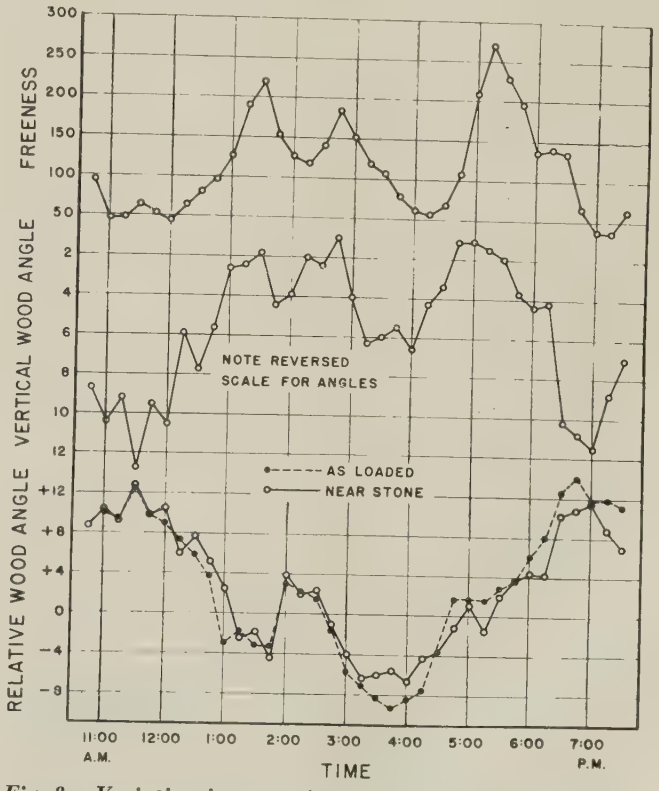

Fig. 8. Variation in a continuous grinder of pit freeness, absolute vertical angle, average vertical angle as loaded, and average vertical angle near the stone during loading at controlled angles

noted times are omitted, the standard deviation of the remainder is only 9.5% of average freeness.

It is of interest to note that the pulpstone used in this test had operated 144 hr. since last previous burring; that it produced pulp of an average freeness of 229 over the test period, and that, three hours after the test ended and the normal, routine mode of loading was resumed, the stone had to be sharpened because both freeness and production rate were too low.

The grinders on which these tests were run are grouped in pairs as shown in Fig. 3. As a convention in measuring vertical wood angle, those sticks which sloped downwardly toward the space between the pair of grinders were said to have a positive angle: sloping the other way, the angle was called negative. Invariably, with routine loading, the average angle was positive although occasional single logs might have negative angles. The angles cited in Figs. 1 to 7 are all positive in the above sense.

It is reasonable to expect that positive and negative angles will have similar effects on freeness. Thus, if half the logs in a given lot had a positive angle of 5° while the balance had a negative angle of 5°, the blended freeness of the pulp made of all the logs would be that corresponding, not to a zero average angle, but to an average of 5°. The angle figures given in the graphs are, therefore, averages of the absolute values of angles for the individual logs, the plus and minus signs of the angles being ignored in the averaging.

A direct check on the validity of this assumption was made in a further test with controlled grinder loading. The results are shown in Fig. 8. On this chart the lower two curves show the angles averaged as usual by ignoring the sign; however, the average so obtained is plotted to indicate whether positive or negative values predominated. The "As Loaded" line shows the average angle of all logs immediately after placing them in the magazine; the "Near Stone" refers to angles measured just above the level of the stone. The differences can be assigned to relative movement of the logs in travel down the magazine; to inability to measure more than half the logs at the lower position; and to errors in measurement. Obviously, from the evidence of the curves, the angle at which logs are loaded into the magazine largely determines the angle at which they are ground.

The central line on the chart gives average angles as positive values only and, as before, the general trend of these angles is in good agreement with the general trend of the freenesses, graphed in the upper line. The increasingly negative angle between 2:30 and 4:00 p.m., has caused a freeness drop qualitatively similar to that produced by the increasingly positive angle between 5:15 and 7:00 p.m. The assumption that the two directions of angle are equivalent seems justified.

Statistical correlation of wood angle and pit freeness measured on a continuous grinder in normal operation strongly suggested, as a major cause of the very large freeness variation found, changes in the vertical angle between wood and stone surface. Confirmation of the effect was sought by varying the angle of the wood as loaded. Freeness variations in the expected directions and magnitudes resulted. There thus seems adequate proof of the validity and importance of the vertical angle effect.

SOURCE OF VERTICAL ANGLE IN THE MAGAZINE

Three factors account for the vertical angle of the wood in the magazines of continuous grinders. These are:

1. The natural taper of the tree. On the spruce-balsam wood used this averaged 0.5-in. diam. difference between ends of 49-in. logs having an average diameter of about 6-in.

2. The several stages of wood conveyors are processes which have varying degrees of bias in selecting whether given logs shall finally be presented with their larger or with their smaller ends closer to the magazine. The man loading

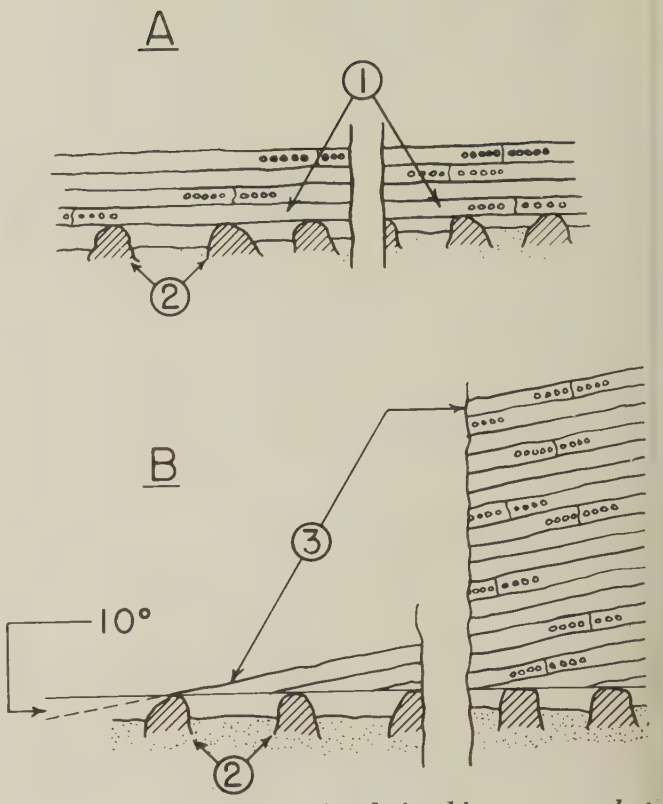

Fig. 9. Wood fiber-stone grit relationship at zero and at moderate vertical wood angle

the magazine has an all-too-human bias in favor of putting his picaroon into the nearer end of a log. A bias in the system which brings wood to the loader with a majority of the smaller ends more readily accessible to him will result in a majority of small ends at one side of the grinder, and a cumulative angling of the wood charge. A check on the loading of a number of grinders showed 47% of the logs so presented that the larger end was first into the magazine. An equal percentage went in small end first, giving a balance. The small residue of 6% also going in small end first built up the positive angle always found in the wood of these grinders under routine loading conditions.

3. The wood fed into a continuous grinder magazine forms, effectively, a continuous, unbroken column over the hours and days. Any small persistent bias in the large end-small end distribution of the wood load will accumulate to the point where the loader finds it intolerable and takes deliberate, corrective action. Further, any angle present will obviously persist indefinitely in the complete absence of bias or of remedial loading.

CAUSE OF THE VERTICAL ANGLE EFFECT

Our knowledge of the grinding process is a very small one at the fundamental level of the exact series of events which lead to separation of a fragment from a block of wood to create a groundwood "fiber." Groundwood "fibers" may cover the broad size range from large aggregates of complete, natural wood fibers to submicroscopic fiber fragments. The specific combinations of interactions among wood, stone grits and water which produce one size limit will obviously not be that which produces the other. The fairly simple overall grinding process which puts together wood, stone, power, and water to give a single blended product is simply the integration of innumerable separate and diverse processes each leading to the freeing of a fragment from the solid billet of wood or, possibly, to the size reduction of previously released fragments.

Despite this innate complexity of the grinding process, a simple theory seems to give a satisfactory account of the very large effect of moderate vertical angles on freeness.

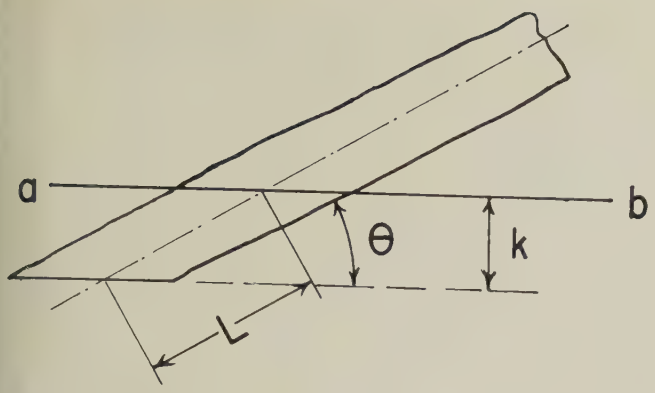

Fig. 10. Idealized sketch of cutting of an inclined fiber by a knife edge

Figure 9A indicates the interface between wood and pulpstone as it is often diagrammed and generally thought of. The fibers lie substantially parallel to the stone surface and the surface fiber (1) is available to and acted on simultaneously by the whole line of grits (2). Under these conditions it would be expected that the fiber could readily be torn free and that this might often be done without breaking it. It is not suggested that whole fibers are always, or even frequently, removed in this situation, but only that they may be.

The interface is again indicated in Fig. 9B but with the wood at an angle of 10° to the stone surface. The fibers are shown in their average diameter-length ratio of about 1:100, the center section of their length having been omitted. Considering the fiber (3), it will be seen that its left hand end is in the plane of the stone, but that its right end is buried in the depths of the log and that 100 sin 10°, or almost 18 fibers, must be removed before the upper end is exposed. Under these conditions it is reasonable to expect the stone action to be confined to the chipping off of small fragments from the exposed end of the fiber and the tearing out of relatively short lengths from the buried residue.

In the case of Fig. 9A, a relatively long fibered, free pulp is to be expected. In that of Fig. 9B it would seem impossible to produce such a pulp. A shorter fibered, lower freeness pulp would be predicted, as found experimentally.

Naturally, this highly simplified picture of fiber removal at zero and at fairly high vertical angle does not describe the removal of even a majority of the fibers made in the one case or the other. In both cases the "innumerable separate and diverse processes" are still going on, but they are apparently influenced as indicated by the added and over-riding effect of the vertical angle.

THE FREENESS-VERTICAL ANGLE RELATIONSHIP

It is apparent that vertical wood angle has an important influence on freeness. All the previously known variables—stone grit and pattern, grinding pressure, wood quality, etc.—still exert their effects upon freeness. It is, therefore, not to be expected that there can be any unique, simple equation expressing the effect of varying wood angle on freeness in all cases. A general form of equation which was found satisfactory for angle-freeness correlations was:

$$F = b \operatorname{cosec} \theta + a$$

where a and b are constants peculiar to one set of data, F is freeness, and θ is the average absolute vertical angle of the wood ground to produce the pulp of freeness F. Where the actual value of θ was less than 2°, the angle was arbitrarily taken as being 2°.

The reason for using the cosecant is indicated in Fig. 10 representing a highly idealized picture of the cutting of a fiber by a knife edge a-b having a depth of cut, k. The length of the cut off portion, as measured along its axis, will be k cosec θ. Since some dim, involved connection between fiber length and freeness is usually assumed, this function seemed appropriate. A lower limit for θ is adopted for two reasons:

1. Freeness obviously cannot follow cosec θ to infinity at $\theta = 0°$.
2. At small angles, say 2° or less, the number of fibers intervening between the stone and the upper end of a fiber, such as (3) of Fig. 9B, will be few. These will have been partially ground away and may well have had their lower ends pulled free from the wood surface to expose the full length of fiber (3) to stone action.

Correlation analysis of freeness against cosec θ gave correlation coefficients of 0.69 to 0.94, showing the relationship to be highly significant. Results of such analyses, expressed as indices of correlation (indices of determination), indicated that 48 to 88% of all freeness variance was associated with the corresponding variance of coces θ. Since it is completely unlikely that any agent may be found which could create this "association" by causing parallel variation of both freeness and angle, it is safe to assume that a large portion of the observed freeness variations are directly caused by variations in the vertical wood angle.

As is to be expected, those tests in which the angle varied most widely gave the highest correlation between cosec θ and freeness by reducing the relative importance of other factors. One factor contributing to scatter is the lack of precision in measuring wood angle: difficulty in knowing just how the axis of a log lay, and inability to measure more than about half of the logs ground, contributed to error in the recorded angles. Figure 11 shows the scatter of points and the trend line of freeness on cosec θ for the test data of Fig. 8.

VERTICAL ANGLE EFFECT IN OTHER TYPES OF GRINDERS

The vertical angle effect may be of importance in any continuous magazine grinder, but is ordinarily of minor importance in other types. The hydraulic magazine grinder is continuous only as regards the magazine. A large wood angle may accumulate in the magazine, and possibly cause transport problems, but each recharging of the actual grinding pocket places the wood on the bare pocket floor, removing any angle accumulated in the magazine. Furthermore, in hydraulic grinders the angle which affects freeness is the horizontal angle of the wood, i.e., the angle of the wood to planes perpendicular to the direction of travel of the pressure foot. Any such angles resulting from disorder in the charge as dropped into the grinding pocket will be minimized or

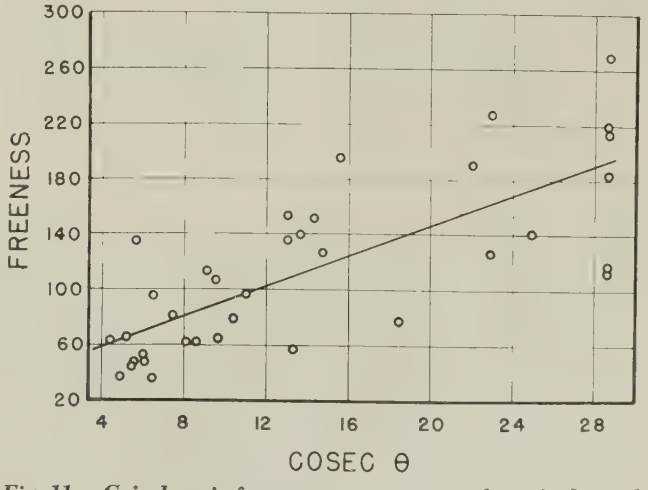

Fig. 11. Grinder pit freeness vs. cosecant of vertical wood angle

Table II. Properties of Pulps Ground at Low and at Medium Wood Angles

	Low wood angle	*Medium wood angle*
Average absolute angle, degrees	1.6	4.8
Freeness		
Unscreened, C.S.F.	241	59
Screened, C.S.F.	228	61
Screenings, %	3.6	2.5
Burst factor	9.8	18.1
Tear factor	55.5	49.8
Breaking length, m.	2310	3520
Bulk, cc./g.	2.57	2.17
Porosity, sec.; Gurley-Hill SPS	59	76
Wet strength ratio[a]	0.450	0.597
Wet breaking length[b]; m. of o.d. sample	66	81
Wet stretch, %[a]	5.2	6.1

[a] Method of R. de Montigny (*4*).
[b] Calculated from wet burst and stretch.

removed as the wood is pressed between the parallel faces of the pressure foot and the stone.

Tests on freeness variations of several hydraulic magazine grinders, again using 15-min. composite samples over 5 to 6-hr. periods, gave standard deviations of freeness averaging 18.2% of average freeness. This is a far better performance than is suggested by comparison with the comparable figure of 37.4% for the continuous grinders. In fact, if all variation could be removed from the hydraulic grinders to give 0% standard deviation, removal of an equal amount of variation from the continuous grinders would still leave a standard deviation of 33% of average. Figure 12 exhibits a representative series of freenesses on hydraulic grinder pit stock.

No means were available for measuring the angles at which the wood approached the stone in the hydraulic grinders. The construction of the grinders seems to ensure that the pertinent angle will remain fairly small and stable. The possibility of significant freeness variation in other types of grinders, due to the angle effect may be estimated from the possibility of having a variable angle of the wood at the stone surface.

ANGLE EFFECT AND GENERAL GRINDING THEORY

The grinding process is sometimes regarded as one in which a considerable proportion of the wood is primarily removed from the log in fairly large fragments, followed by size reduction and refining of these fragments in passage across the stone-wood interface. The effect of wood angle reduces the chance that this is the major mechanism.

If varying wood angle produces its effects through the general mechanism described with reference to Fig. 9, its effect is then principally concerned with the primary removal of fibers from the solid wood. Any theory that gave to regrinding of separated fiber a major role in determining product quality would require that the regrinding process too should, by some unknown mechanism, also be strongly affected by wood angle. Such a mechanism may exist, but it seems more plausible to assign the greater part of the angle

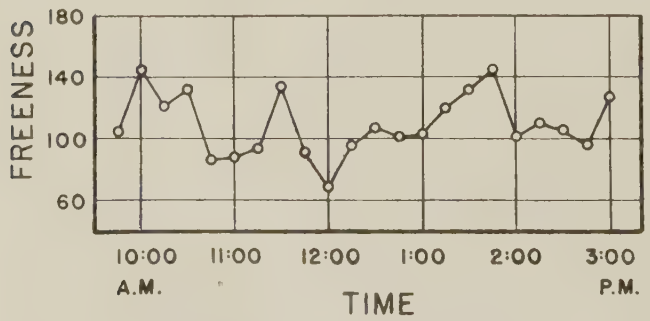

Fig. 12. Variation of pit freeness in normal operation of a hydraulic magazine grinder

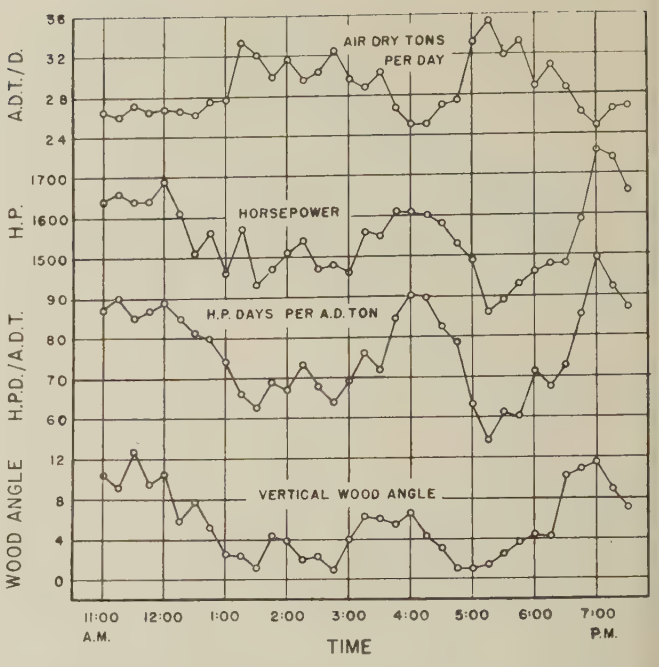

Fig. 13. Variation of grinder motor load, grinding rate, unit power per ton, and vertical wood angle; from same test as data shown in Fig. 8

effect to its action in the actual moments of separation of the "fibers" from the solid wood.

Angle Effect on General Groundwood Properties

The angle effect has been defined largely with reference to pulp freeness to minimize the required testing labor. It is understood that freeness only stands surrogate to all the other properties of interest as probably the best, but by no means a complete index of groundwood quality. The general properties of high and low freeness groundwoods, where the freeness change is predominantly due to change in wood angle, are given in Table II. The high freeness pulp (low angle) is a composite of the samples made at 5:00, 5:15, and 5:30 p.m. (Fig. 8), while the low freeness values are for 4:00 to 4:30 p.m. The available data are too scanty to permit a definite statement on the point, but it appears that the other properties associated with the high and the low freeness pulps are much as would be expected were the freeness changes made by operation of more familiar controls.

Effect of Wood Angle on Grinder Load and Production Rate

Changes in wood angle produce small but definite changes in the power required for grinding. In the test of Fig. 8 the effect was a 20-hp. increase per 1° increase in wood angle. The percentage change was about 1% per degree.

In the same case, grinding rate was somewhat more affected by wood angle, having an indicated change of about 2% decrease per degree increase in angle.

Power input per ton of groundwood increased over 2 hp.-days per air-dry ton for each degree increase in wood angle. The spread of this factor between highest and lowest angles in the same case was 24 hp.-day per air-dry ton. Figure 13 shows motor load, grinding rate, and unit power together with wood angle for this particular test.

Incidentally, a second mechanism which may cause uncontrolled drop in freeness and rise in unit power input is partial jamming of the wood in the magazine with consequent reduction of the effective grinding pressure. Such incidents are readily distinguished from those directly due to wood angle. In the former case, although unit power per ton increases, total power decreases. With increasing wood angle both total power load and unit power per ton increase.

MINIMIZING THE WOOD ANGLE EFFECT

The range of freeness variation which may result from a fairly wide range of wood angle, say 0° to 10°, is much larger than can readily be counterbalanced by use of the usual grinder controls. When angle is high, and freeness much too low, it is theoretically possible to raise freeness as desired by grinding faster. Actually the limits of wood feeding force available, and the maximum power load which can be drawn, severely limit the scope of this remedy.

The most practical corrective is better loading of the magazine, obtainable through operator training in the importance of careful loading, plus good supervision. Mechanical magazine loading systems, more of which will be seen in the future, will require randomizing of the positions of the large and small ends of the logs. This may be had by end-for-end reversal of the position of every second log just prior to loading.

CONCLUSION

It is believed that an important but hitherto overlooked factor in groundwood quality variation has been found. The results reported were obtained by test work on commercial grinders and suffer from the difficulties and the reduced precision often attending work done under these conditions. The wood angle factor is a very potent one in its control of product quality and would seem to merit more detailed investigation than could be given it in this project.

Further investigation of this factor should make a rewarding subject for study on a miniature grinder. Actual grain angle of the wood ground could be selected and measured. Wood at varying grain angles could readily be had—matched for moisture, density, and the other factors which varied in unknown degree in the present work. With these factors stabilized further studies of the effect of wood angle may help in elucidating the fundamentals of the groundwood process.

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Thank you, Mr. Beath. Are there any questions? Here is one over here.

K. G. Booth (Abitibi): Mr. Beath, did you happen to have a chance to notice any difference in the opacity between pulps ground at normal and fairly high angles?

Mr. Beath: No. We made no tests on that. The work done involved a lengthy series of tests and a large number of samples, and we confined ourselves amost entirely to freeness to permit making more tests while minimizing the lab work. We were primarily concerned with the freeness variations in our production, so we felt that if we could eliminate those we could eliminate all the other variations that accompany them.

Mr. Maune (International Paper): Do you find any difference in the species of wood that is put through as in poplar at an angle.

Mr. Beath: We used run-of-the-mill wood, which is a spruce-balsam mixture. We made no effort to check on changes in the relative proportions of the species. I suspect that a good deal of the residual variation that is not explained by angle is due to the nature of the wood, either as to species, density, or soundness, but we have not checked on that point.

A. J. Pearson (Australian Newsprint Mills Ltd.): Mr. Beath, could you tell us if wood should be ground parallel to or at an angle to the grain for optimum strength. I noticed that your strength results showed higher values when the wood was deliberately ground at an angle but the freeness was lower. If you had reduced the freeness to this same value with the fibers parallel to the stone, would there have been any difference in strength?

Mr. Beath: We do not have the evidence on which to base a sound answer to that, but it was our general observation that the freeness changes produced by angle changes seemed to be accompanied by changes in the other qualities which were just such as you would expect if you had changed the freeness by dulling stones or reducing grinding rate. I can't give you that as a conclusion. It is highly tentative. We didn't do enough work on strength properties to provide a reliable answer to your question.

Mr. McCracken (Provincial Paper): I would like to ask Mr. Beath, if in his work (I presume he did no fiber classifications) he feels that in parallel grinding, with perfect loading conditions, there was a much higher percentage of longs with a corresponding decrease in fines?

Mr. Beath: I would expect that to be the case from the freeness trends. The stones in the grinders on which these tests were run are conditioned to run at a moderate wood angle, because that seems to be the normal loading condition. In other words, if we put a level load of wood in we invariably got a freeness very much higher than we wanted, and I would assume that we had a high fraction of longs and a reduced proportion of fine fiber.

Mr. McCulloch (Consolidated): This isn't in the nature of a question but the meeting may be interested in our experiences along that line. We have continuous grinders which are loaded at the rate of 3.4 cords per man-hr. which is quite a high rate and we do experience some of the trouble described by Mr. Beath. We have always thought that poor loading was almost completely responsible for off-quality stock and freeness variations. Lately, however, we have been experiencing difficulty with worn-out wear strips and chains. These are currently being reconditioned. The effect on the grinder is to give a sliding rather than a rolling friction as the chain advances. This result in erratic load control.

After reconditioning our wear strips and installing a new set of chains on one of the grinders, tests revealed that our short-term pit freeness variations were greatly reduced. We run composite freeness tests on each grinder every 4 hr. as a routine control test, so observations over a long period before and after the change could easily be made.

It appears that with inadequate wear strips and chains one chain may have a tendency to chew through one end of the log while the other chain maintains its grip. This in turn causes jamming and possible tipping close to the stone. The net result is similar to poorly loaded wood.

In order to remedy this situation we reverse our chains and remove the "chewed-up" wood, through the door. Sharpening is only used as a last resort since we feel that by sharpening, the pulp will be degraded after the jammed wood is cleared from the magazine.

Mr. Perry: Do you think that the angle set up when the stone is sharpened from only one side would be influential? This difference from one side of a stone to the other amounts to perhaps a quarter of an inch or more in diameter, with perhaps a 3° angle.

Mr. Beath: No, I think not. I think it would be too small to have very much effect. Angles of the order of 1 or 2° have relatively small effect.

Mr. Brauns: When I observed the title of this exceedingly interesting paper in the program it aroused my curiosity and caused us to run hurriedly a few experiments on our small grinder to supply some information for our own benefit. I have just received some results by mail, and hope that Mr. Beath does not mind if I report them here at his own show.

We cut square blocks out of a number of logs at various angles to the log axis, and in a way so as to insure that all logs were equally represented in one run. The runs were numbered at random in order to avoid misinterpretation of the results due to changes of the stone surface. At angles above 20° the freeness test did not work, but let me report the following values:

Angle	*Freeness*	*Strength*	*Bulk*
0	160	2420	2.48
5	69	3300	2.3
10	25	4400	2.0

I may call your attention to the fact that the log freeness-strength relation is a straight line (a Paterson curve) and that it runs parallel to similar lines produced by changes in the stone surface; in other words, a change in the angle between the wood axis and the stone surface has exactly the same effect as a change in the stone surface condition. We are continuing these experiments as Mr. Beath has suggested should be done on an experimental grinder.

Mr. Beath: I would like to thank Mr. Brauns for his contribution. I was hoping someone who had a miniature grinder would investigate the point with more precision than we were able to do with commercial grinders.

J. F. Natwick (Consolidated Water Power & Paper Co.): I am a little curious about what you would do if you had con-

siderable crooked wood. It would automatically set up varying degrees of angle if you were trying to load that wood. Would you feel that it would be necessary to throw it out, or, if you did throw if out would you then load nothing but crooked wood in one of your grinders, then you would have a pulp, I suppose, of a lower freeness. How would you handle that if your are definitely trying to load your grinders the way you indicated?

MR. BEATH: I don't know what the answer to that is. It seems to me to require an economic answer regarding the value of your crooked wood against the disturbance it may cause to your quality standards. I can't say that each log is ground to a specific freeness, that depends upon its angle, but the freeness obtained from any log is affected by the angle and the final pulp freeness is a blended average. The angle effect is a blended average also of the angles of the individual logs, so that a single crooked log in a dozen straight ones might not be important. If they were all crooked I think it is an economical question as to whether you wish to use them or not.

MR. ATACK (Pulp and Paper Research Institute): Mr. Brauns, May I inquire if the data you have presented were obtained under conditions of equivalent loading on the grinder?

MR. BRAUN: Yes, we maintained the same dead load on the grinder, the stone was well conditioned for several hours and each run lasted about 6 min. The grinding pressure was about 30 p.s.i. on the actual wood surface, the temperature 60°C., and the peripheral speed 18 m. per sec. The energy consumption was 980, 1050, 1670 kw.-hr. per ton for the respective runs.

MR. CURTIS (Australian Newsprint Mills Ltd.): Mr. Beath, I think that some of those present who have visited Boyer and seen some of the very large trees that we deal with, and the problems that compression and tension wood might present, would be interested in our experience. I think Mr. Pearson might have some figures available of the degree of variation that we get with the eucalypt wood that we use.

MR. PEARSON: For the benefit of those who have not been to our mill, we use for groundwood, very large eucalypt trees. We cut sawn billets 7 by 7 in. by 4 ft. for grinding in Tidmarsh and Great Northern grinders. In some research work we did in the mill for a completely different purpose, we selected carefully billets in which the fibers were parallel to the sawn surface and ground these on a Great Northern grinder in such a way that the fibers would be all parallel to the stone. We tested the pulp from the grinder at 3-min. intervals and found a freeness range from 40 to 140 C.S.F. Thus we can obtain a wide range in freeness when the wood is ground with fibers parallel to the stone.

MR. NATWICK: These remarks on these varying freenesses point up the fact that the groundwood mill will inevitably produce pulp of greatly varying freeness. The irregularities may be nearly completely eliminated by large blending or tempering tanks between the pulp mill and the paper machines.

MR. MCCULLOCH: In answer to this gentleman's inquiry about tanks, at Port Alfred we are blessed with a couple of great big tanks which are in series with our system and they, no doubt, help to take care of a lot of variations. I would like to ask Mr. Beath a question. That is, what percentage of your trouble would you attribute to long wood? Do you have any long wood problems?

MR. BEATH: Well, the specific variation we were looking for, and had present in our grinder, as far as I know had very little connection with long wood. You are referring to wood which actually jams and stalls the chain or slows it down drastically and causes freeness change?

MR. MCCULLOCH: I was referring to a log that may become jammed in the magazine causing some of the other logs to tip on the way down.

MR. BEATH: I didn't mention it, but we did measure angles of wood, as loaded, on individual logs. The logs were end marked to identify them and the angles were again measured just above the level of the stone, so that we have records of the history of the individual logs and what happened to them going down the grinder magazine. As one of the charts showed, the average angle at the stone is almost entirely due to the average angle of the wood as loaded. However, so far as individual sticks are concerned, we would occasionally find quite a large change in angle in its course down the grinder, but the number of sticks which did change their angle to any marked degree was so small that it had very little effect on the average.

MR. MCCULLOCH: I imagine that with experimental work you were quite careful not to load long bolts into the magazine. I am just wondering if, aside from your experimental work you discovered any effect.

MR. BEATH: Well, apart from the test in which we actually controlled the angle of the wood as we wished it to be, whether we wanted it level or at some other angle, our tests relating freeness and angle were done without reference to loading. The loading was completely normal and we didn't appear on the loading floor to give the loaders instructions. I doubt if they had a clue as to what was going on, but I don't think we have much trouble with over-length wood. We have over-length wood, of course, but they throw it out before it goes into the magazine.

Effect of Groundwood Pulp Quality on Wet End Efficiency of a Paper Machine

GUENTER HERWIG

THE efficiency of the wet end of a paper machine is determined by mechanical conditions, drainage capacity of the machine, formation, basis weight of the paper being produced, and drainage and wet strength properties of the pulp.

While the control of the above first four factors is the task of the machine room, drainage properties and wet strength of the pulp must be controlled in the groundwood mill. Control tests must evaluate the quality of the pulp rapidly and under conditions as they prevail at the wet end of the paper machine.

It was standard practice in this mill to control groundwood pulp quality by testing freeness and bursting strength of the pulp. Experience showed, however, that predicted effects of these two pulp properties too frequently contradicted actual effects. The reasons for these discrepancies were, obviously, in the very nature of the tests.

The freeness test indicates the speed of drainage of the pulp on the table roll part of the paper machine, but it does not furnish information concerning secondary drainage on the suction boxes and suction couch. This secondary drainage determines the moisture content of the wet web between the couch and the first press. It is this moisture content which, in turn, determines the wet strength of the web. On the other hand, bursting strength of the groundwood pulp does not relate to wet strength. It is therefore useless to the control of pulp properties affecting the efficiency of the wet end.

Realizing the limitations of freeness tests as a means of evaluating groundwood pulp quality, a new method was introduced. The objective of the work reported here has been to determine the reliability of this method in predicting paper machine performance.

NEW METHOD

This method was developed by Brecht and Klemm (*1*). It permits the evaluation of primary and secondary drainage properties of pulps under conditions similar to those on the table rolls, suction boxes, and suction couch. The drainage tests are performed while forming a groundwood sheet for the wet strength test. The whole operation, from the preparation of the pulp sample to computation of the results, requires 25 to 30 min.

Instruments and Testing Procedure

The drainage properties of the pulp are determined in the German standard sheet mold "Rapid Koethen." The apparatus is fitted with a pump which allows the evacuation of the suction chamber below the sheet-forming wire.

About 2.36 g. ovendry pulp are used to form a wet strength test sheet with a basis weight of 75 g. per 50 sq. m. = 50.6 lb. (25 × 38—500). The initial consistency of the pulp suspension is approximately 0.34 g.p.l. At the moment the suction chamber is being evacuated, dilution water begins to drain through the forming fiber mat and wire into the suction chamber.

The end point of the increasing vacuum is reached at the moment the water mirror on the sheet disappears. This end point depends on the drainage resistance of the forming fiber mat. The higher the drainage resistance, the longer the time required to drain the water, and the higher the vacuum in the suction chamber (Fig 1). This vacuum is designated V_1.

A second vacuum V_2 is established at the moment air enters the suction chamber after the removal of the dilution water. The amount of air entering the suction chamber depends on the air porosity of the wet fiber mat. The higher the air

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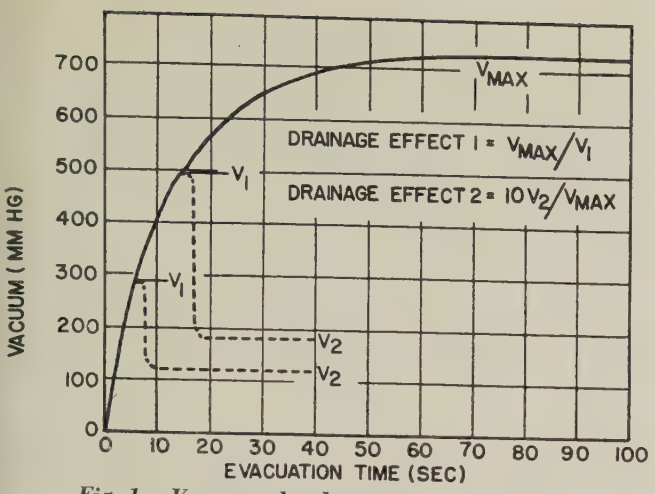

Fig. 1. Vacuum development in sheet mold

porosity, the more air can enter and the lower the second vacuum will be (Fig. 1).

After 10 sec. of drawing air through the sheet, 3 strips of 3 cm. width are removed for wet strength tests on the Darmstadt wet strength tester.

The vacuums V_1 and V_2 are then made independent from the pump capacity by setting them in relation to the maximum vacuum obtained after 60 sec. evacuation of the suction chamber, sealed air tight. The computed values are then called Drainage Effect 1 and Drainage Effect 2 (Fig. 1).

$$\text{Drainage Effect 1} = \frac{V_{\text{Max}}}{V_1} = D_2$$

$$\text{Drainage Effect 2} = \frac{V_2}{V_{\text{Max}}} = D_2$$

The ovendry content of the test sheet can then be calculated by using these two values.

$$\text{Combined drainage} = K\,(10 + D_2 - \sqrt{D_1})\ \%\ \text{B.D.} \qquad (K = 1.45)$$

Thus, the initial wet strength can be corrected to a standard bone-dry content of the test sheets.

Brecht and Klemm demonstrated the relationship between Drainage Effect 1, Drainage Effect 2, and initial wet strength, and how these values can be used to interpret groundwood pulp quality (*1, 2*).

Drainage Effect 1

Drainage Effect 1 depends on the amount of fines present in the pulp, regardless of the quality of the fines. Decreasing the amount of fines increases Drainage Effect 1, and vice versa. Increasing values of Drainage Effect 1 point to an increasing drainage of the pulp.

Drainage Effect 2

Drainage Effect 2 depends on the amount of fines present in the pulp as well as the character of the fines themselves. Thus Drainage Effect 2 is partially dependent on Drainage Effect 1. Drainage Effect 2 decreases with increasing Drainage Effect 1, and vice versa. Relatively high values of Drainage Effect 2 in relation to Drainage Effect 1 indicate that the fines are mostly fibrils, while relatively low values point to a predominance of flour fines (Fig. 2).

Initial Wet Strength

This pulp property depends not only on the proportional amount of fibers and fines of the pulp, but also on the quality of the constituents (Figs. 2 and 3). Neither groundwood fibers nor groundwood fines alone develop wet strength. Only the mixture of the two develop wet strength.

Optimum conditions exist when the voids between the fibers are filled with fines. At this point of saturation the wet strength is high, and Drainage Effect 1 and Drainage Effect 2 are relatively high. If the groundwood fibers happen to be predominantly slender and long in character, a minimum amount of fines will be required to fill the voids. This will favor Drainage Effect 1. Due to the greater plasticity of this type of fiber, Drainage Effect 2 will be higher, too. This in turn favors the already high wet strength because the increased density results in a sheet with higher ovendry content (Fig. 1).

Shorter, chunkier groundwood fibers require many more fines for saturation. Therefore, Drainage Effect 1 will decrease and the wet strength of this type of pulp will be lower due to the poor felting characteristics of the short chunky fibers.

A shift from the point of saturation will in either case decrease the wet strength of the pulp and will be accompanied by changes of Drainage Effect 1 and Drainage Effect 2. For example, increasing the fines content slowly beyond the amount of fines required for saturation will slowly decrease the wet strength and Drainage Effect 1 and increase Drainage Effect 2.

At extremely high fines content, Drainage Effect 1 will be extremely low. The wet strength will approach zero and offset the gain of the higher ovendry content of the sheet caused by the increase of Drainage Effect 2.

A shift away from the point of saturation toward a higher fiber content will increase Drainage Effect 1. But, at the same time, the wet strength will decrease. Since Drainage Effect 2 will be lowered, the moisture content of the sheet increases, which results in an additional decrease of wet strength.

PRELIMINARY INVESTIGATION

Multiple correlation techniques (*3, 4*) were used to test the significance of the three variables, Drainage Effect 1, Drainage Effect 2, and initial wet strength, of groundwood pulps on the wet-end efficiency of No. 2 machine. Tour averages of groundwood test data for the month of June, 1957, were correlated with the paper machine efficiencies of the corresponding shifts, excluding as far as possible all side effects not attributable to groundwood pulp properties.

The results of this preliminary investigation indicated a

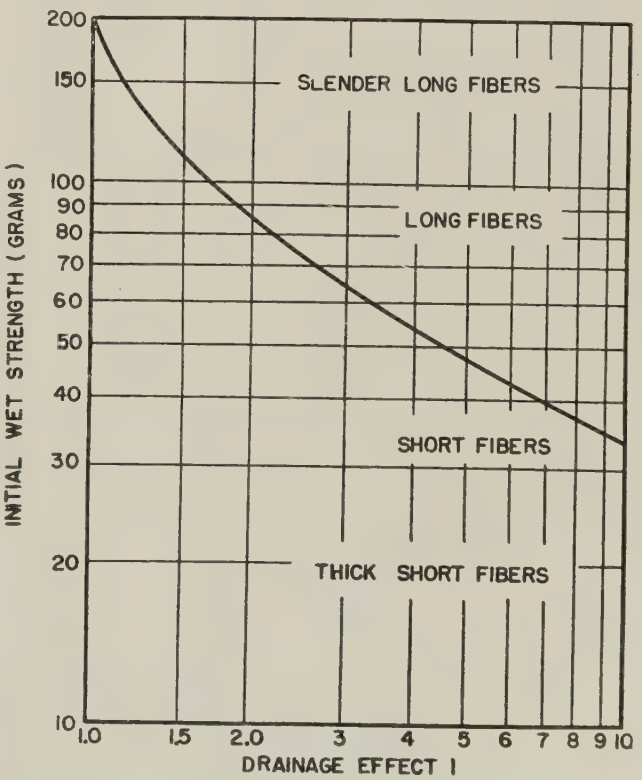

Fig. 2. Effect of fiber structure on initial wet strength

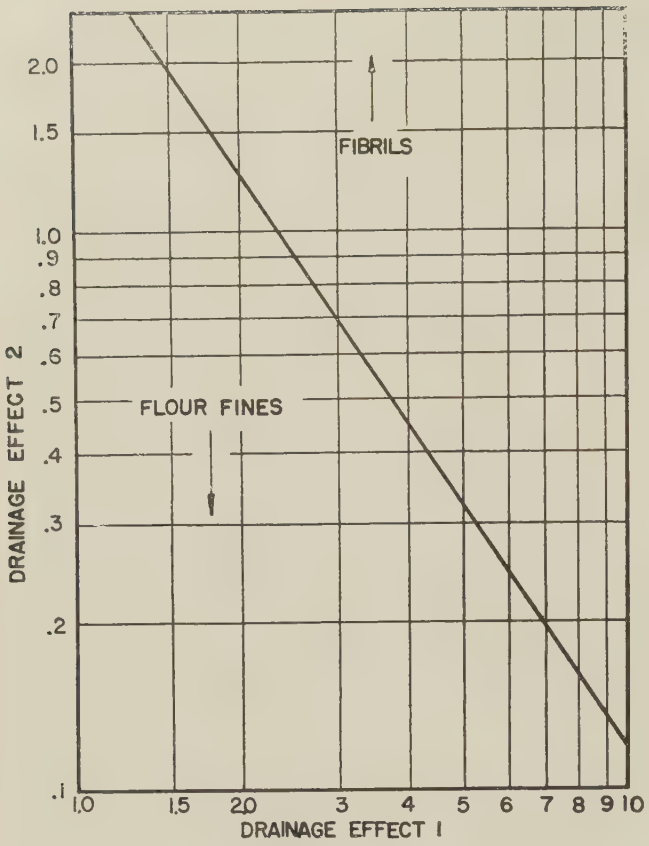

Fig. 3. Effect of fibrils and flour fines on drainage effect 2

significant effect of initial wet strength on the wet end performance. However, Drainage Effects 1 and 2 were tested insignificant due to large residual variations. Including basis weight, ash content, amount of broke, amount of chemical pulp, headbox temperature, and paper machine speed as additional independent variables in the multiple correlation, these reduced the residual variations to a point where Drainage Effect 1 and Drainage Effect 2 became significant as independent variables.

Preparation of Data

Nine-factor multiple correlation involves a heavy amount of arithmetic. It was, therefore, decided to correlate 10-day moving averages of test data with 10-day moving averages of paper machine wet-end efficiencies to bring the number of variables down to 3. It was assumed that the other variables would become constant as 10-day moving averages. If not, this would just inflate the residual variance and thus aggravate the test of significance of Drainage Effect 1, Drainage Effect 2, and initial wet strength.

The correlated data covered the period from July 9, 1957, through Dec. 31, 1957.

The 10-day moving averages of Drainage Effect 1 ranged from 1.4 to 1.9 Drainage Effect 2 from 2.2 to 1.3, initial wet strength from 33 to 73 g. per cm.

The over-all efficiency of the wet end varied between 82.5 and 97.1%, efficiency gains or losses due to speed changes between +1.9 and −7.9%, and efficiency losses caused by wet-end breaks between 2.1 and 15.0%.

RESULTS

The multiple correlation equations fitting best the data were:

1. Efficiency gain or loss due to changing speed = E_S

$$\% E_S = -9.9 + 4.5 \times D_1 - 4.4 \times D_2 + 0.18 \times IW$$

2. Efficiency loss due to wet-end breaks = E_B

$$\% E_B = -38.4 + 25.0 \times D_1 + 13.42 \times D_2 - 0.37 \times IW$$

3. Overall efficiency = OE

$$\% OE = +111.4 - 13.0 \times D_1 - 13.7 \times D_2 + 0.52 \times IW$$

The significance of the correlation equations were tested at better than 99.9%.

The corresponding precisions of the predicted values were for:

$$E_S \pm 3.3\%$$
$$E_B \pm 4.1\%$$
$$OE \pm 7.2\%$$

The isolated effects on E_S, E_B, and OE of Drainage Effect 1, Drainage Effect 2, and wet strength as independent variables were significantly better than 99%.

Influence of Drainage Effect 1, Drainage Effect 2, and Wet Strength on Paper Machine Efficiency

The dependency of the variables and their influence on the paper machine is demonstrated in the following two examples. An average Drainage Effect 1, Drainage Effect 2, and wet strength is selected as a starting point for the calculation of the paper machine efficiency.

Example 1:

Average Drainage Effect 1 = 1.6
Average Drainage Effect 2 = 1.97
Average wet strength = 53 g. per cm.

Substituting these values in the regression equations, the paper machine efficiencies are calculated as follows:

$$E_S = -1.7\%$$
$$E_B = 8.7\%$$
$$OE = 91.2\%$$

Assuming that the groundwood pulp should be freer to meet the drainage capacity of the paper machine, Drainage Effect 1 has to be increased. Increasing Drainage Effect 1 from 1.6 to 1.9 decreases Drainage Effect 2 from 1.97 to 1.35 and decreases the wet strength from 53 to 35 g. per cm.

The new efficiencies would be:

$$E_S = -1.0\%$$
$$E_B = 14.4\%$$
$$OE = 87.4\%$$

It can be seen that the increase of Drainage Effect 1 from 1.6 to 1.9 improves slightly the paper machine efficiency loss due to the speed change. On the other hand, the drop of wet strength from 53 to 35 g. per cm. increases the efficiency losses caused by breaks from 8.7 to 14.4%. This increased loss of paper machine efficiency eliminates the little gain made by increasing the paper machine speed. As a result of this, the over-all efficiency drops from 91.2 to 87.4%.

Example 2. Realizing the over-all importance of wet strength on paper machine operation, one might want to increase this wet strength by producing a groundwood pulp with lower Drainage Effect 1.

Decreasing Drainage Effect 1 from 1.6 to 1.4 increases Drainage Effect 2 from 1.97 to 2.2 and increases the wet strength from 53 to 65.

This time the efficiencies would be:

$$E_S = -1.2\%$$
$$E_B = 2.4\%$$
$$OE = 96.8\%$$

By decreasing Drainage Effect 1, a loss of efficiency might be expected. However, wet strength and Drainage Effect 2 are relatively high. Therefore, the effect would be that they maintain the efficiency loss at a level experienced at the higher Drainage Effect 1 of 1.9 in the previous example. The beneficial effect of the higher wet strength is reflected on the relatively low efficiency loss of 2.4% caused by breaks. The over-all efficiency of 96.8% is much higher than any of the two previous examples.

CONCLUSIONS

It has been shown that groundwood pulp quality, as defined by Drainage Effect 1, Drainage Effect 2, and wet strength has a considerable effect on paper machine performance. Each factor was proved statistically significant. Testing groundwood pulps for Drainage Effect 1, Drainage Effect 2, and wet strength, allows one to anticipate paper machine performance well ahead of the time the groundwood pulp reaches the machine. The ease and speed of this testing method makes it superior to all other methods known.

Our experience showed that it is extremely difficult to change independently any one of the variables from the other, especially wet strength characteristics. Realizing this, one should strive for a balance between Drainage Effect 1, Drainage Effect 2, and wet strength, in order to maintain high paper machine efficiencies.

Management and operating personnel of our mill have found confidence and reliability in the new testing method. Today we apply it not only to the control of the groundwood mill but also to the control of pulp preparation in general.

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Fundamentals of Groundwood Evaluation—A New Approach

JAMES d'A. CLARK

It is self-evident that if enough were known about a physical and structural characteristics of the individual fibers in a sample of pulp, then the papermaking qualities of that pulp should be predictable with accuracy and certainty. The problem then is to select an optimum set of fundamental fiber characteristics that are (a) as independent as possible of each other, (b) are measurable numerically without undue difficulty, and (c) are adequate in kind and number to constitute a frame of reference sufficiently comprehensive to enable the required papermaking predictions to be made.

One framework that appears to fulfill these requirements comprises: (1) fiber fineness (weight of a fixed length of fibers laid end to end); (2) fiber length (weight-average length); (3) fiber cohesiveness (after pressing and drying together under standardized conditions); (4) intrinsic fiber strength (ultimate breaking stress); (5) flexibility when wet (the compactness or density of a standardized test sheet made from them).

Apart from visual qualities, such as color and cleanliness, it seems probable that only the first three characteristics are needed to define adequately the papermaking qualities of groundwood pulp made from a given wood species. In the case of groundwood, the intrinsic strength of the fibers usually is more than enough to affect appreciably the strength of a sheet of paper made from the pulp. Furthermore the ultimate tensile strengths of the various woods used for groundwood are not very different, particularly when compared on the basis of a common specific gravity, as may be seen in Table I.

Similarly it will be noticed that the green or wet modulus of rupture by bending and the modulus of elasticity E of these woods are quite similar, so that the flexibility of the fibers, which is inversely proportional to E and to I, the moment of inertia when wet, should be almost wholly a function, and a quadratic function at that, of the average fiber fineness.

Commonly employed measures of groundwood pulp quality are (1) freeness, (2) burst, (3) tear, and to a limited extent, (4) specific volume (or its inverse, density, or specific gravity of test sheets).

Freeness measurements have value for the routine control testing of groundwood, and for groundwood, freeness may have some value as a definitive test although a somewhat uncertain one and one most difficult to analyze. Burst and tear factors of course have been very valuable in characterizing groundwood, particularly if the specific volume is also known. Unfortunately the fundamental analyses of both these strength tests with respect to fiber characteristics are also so complex that they are almost impossible to apply in an inductive approach to better grinding methods or better groundwood. By deductive methods and by skillful observation and experiment, the art of grinding wood has progressed favorably during the past half century but causes and effects are still very poorly understood. Moreover the advent of the newer processes such as chemigroundwood and mechanical pulp from chips, has raised new difficulties with the interpretation of the freeness-burst-tear pulp testing system.

It is one of the purposes of this paper to suggest that if the present system were replaced or at least supplemented by the much more fundamental fineness-length-cohesiveness system, a far clearer insight into grinding and the properties of groundwood, would result.

FINENESS

This is a relatively new test (*1*) which was recently improved and revised in TAPPI Suggested Standard T 234 m-58 (*2*). A known concentration of fibers is suspended or dried on a known area of a cell or slide (say about 1 to 2 mg. per 100 sq. cm. of slide area) and projected on a screen having lines of a fixed length in a fixed area. The coarser the fibers in the specimen, the fewer will be the crossings of their images on those lines and if all the line-image crossings are counted, it is possible to calculate the average coarseness of the fibers very simply. The result may be expressed as so many mg. per 100 m. The term "grex"—g. per 10,000 m.—having been proposed as the international denier, the unit of

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Table I. Strength Properties of Pulpwoods

Wood	*Specific gravity, g. dry/cc. air dry/(A)*	*Dry tensile strength, p.s.i. (B)*	*Tensile/ sp. g., (B)/1000 (A)*	*Specific, gravity, g./dry/cc. dry/(C)*	*Modulus of rupture (green), p.s.i.*	*Young's modulus (E) (green), 1000 p.s.i.*	*E./sp.g. (E)/((C) × 10*
Western hemlock	0.40	10,500	26	0.44	6100	1220	28
Loblolly pine	0.48	15,000	31	0.54	7300	1400	26
Eastern spruce (?)	0.39	13,700	32	..	..	..	..
White spruce	..	..	..	0.45	5000	1070	24
Black spruce	..	..	..	0.43	5400	1060	25
Sitka spruce	0.41	11,200	27	0.42	4700	1230	25
Balsam fir	..	..	..	0.41	4900	960	23
Eastern hemlock	..	..	..	0.43	6400	1070	25
Aspen poplar	..	..	..	0.41	5400	1120	27

Source: Technical Bulletin No. 479. U. S. Dept. of Agriculture.

mg. per 100 m., might be called a "decigrex" abbreviated to "dg." Fibers in a fine chemical pulp will test about 10 decigrex or less; in a coarse pulp 20 dg. or more.

Just as with textiles, fineness is a very important property of any paper pulp fibers. It has been almost wholly neglected in the past because it has been so difficult and tedious to measure accurately in micro-amounts with a microscope. Although, because of its newness, there is presently very little correlative data, especially for groundwood; it seems evident that with other factors being equal, the sheet with the more slender fibers will have a higher density, burst, stretch, tensile, fold, smoothness, and a decreased air porosity. The pulp also will be wetter.

Because the flexibility of a rod depends on I which is proportional to the fourth power of its diameter (or, if hollow, on the difference between the fourth power of its diameters), and the modulus of elasticity E of its material, and since all the woods commonly used for groundwood have moduli of elasticity of about the same order, fiber fineness is by far the predominant factor in the density of the resulting test sheets. This predominance is further enhanced because the munbers of fibers in a sheet of a given basis weight are inversely proportional to the square of their average diameter. Campbell (*3*) has pointed out that the initial compacting force in a sheet of wet fibers as the water goes from them, is surface tension, which amounts to about 140 dynes per cm. of fiber and is independent of fiber thickness. Thus by increasing the slenderness, the flexibility is increased by the fourth power, and the compacting initial forces by the second power. It follows that fiber slenderness has a tremendous influence on pulp wetness and on the interesting and important property of test sheet density.

A small increase in fiber fineness at a given sheet density, similarly provides the sheet structure with a greatly increased internal flexibility, enabling the sheet better to distribute and thus resist, any load applied to it, such as a tensile or bursting force, and more especially the complex forces applied during a folding test.

TEST SHEET DENSITY

As described in TAPPI Standards T 205 m and T 220 m, this is measured as g. per cc. (or its inverse, specific volume in cc. per g.) of standard test sheets.

It is a pity that there is not a quicker test available for measuring specific volume. A possibility is to weigh the mercury displaced by a portion of an ovendried, thin pad of pulp.

The density of an uncalendered sheet of paper made under constant pressing conditions, has a profound influence on the strength and other properties of the sheet and is also believed to be the best available measure of the true wetness of the pulp on a paper machine.

The main property leading to increased sheet density is fiber flexibility when wet, which is dominated by fiber slenderness, as just discussed. Also affecting sheet density are the initial compacting forces and the plasticity of the fibers, both of which are influenced greatly by fibrillation, macro- and micro-, probably down to fibrils of molecular dimensions.

The author has shown (*4*) that the tensile strength, burst, and rigidity factor E of standard test sheets, are directly proportional to their density, and the lateral absorbency and tear factors inversely so. In brief, this is because the fibers in a more compact sheet are more firmly pressed together and consequently it takes more force to separate them. On the other hand, a tearing force applied to a more compact sheet is concentrated on a smaller resisting zone at the apex of the tear and so the structure is more readily broken. Fiber length has a beneficial influence on tear because it serves to expand the area of the stressed zone which area may be expanded also by finer fibers because of their greater flexibility.

FIBER LENGTH

This is the weight-average fiber length as determined by a classifier, calibrated and the results calculated as described in TAPPI Standards T 232 m and T 233 m.

If one mixed 100 lb. of a long fibered pulp A having a uniform fiber length of 5 mm. with 5 lb. of very short fibers B each with a uniform length of 0.25 mm., since there is the same number of fibers in the 100 lb. of A as there is in the 5 lb. of B, the number-average fiber length of the mixture would be approximately 2.6 mm. In other words the addition of 5% by weight of white-water fines, has apparently reduced the average fiber length of 100 lb. of a select pulp, as reckoned by the number or arithmetical average, by nearly one-half. This is nonsense. Actually the effective fiber length of the long pulp would be reduced only by about 5% as the calculated weight-average length of the mixture would show.

The author has reported (*5*) the following experimental results using fibers that differ only in length and not at the same time in fineness. Test specimens of the pulps were prepared by classifying a number of batches of bleached sulfite pulp, discarding all the short (and fine) fractions, making the long fractions into pads, drying, cutting up the pads into very thin strips with a razor, then reclassifying. Viscose rayon similarly cut to various lengths was also used to confirm parts of the work. He found:

1. The tensile strength and Young's Modulus E of a test sheet, other things being equal, were proportional to the square root of the weight-average fiber length, i.e., to $\sqrt{L}$, the burst was directly proportional to L, and the tear was proportional to $\sqrt{L^3}$.

These findings were confirmed at least in part by some work done at the U. S. Forest Products Laboratory (*6*) and also are in good agreement with the findings of some recent work done by the Institute of Paper Chemistry (*7*).

2. When a portion of a long fibered, gently beaten sulfite pulp C which had a fairly uniform average fiber length of 3.08 mm. was mixed with an equal weight of a short-fibered fraction from the same pulp of the same coarseness but having a fairly uniform average length of 0.54 mm., as may be seen in Table II, the resultant mixture of long and short fibers having a weight-average length of 1.81 mm. gave sheets with the same tensile strength as pulp D with uniformly long fibers of about that length, but 30% higher than the uniformly short pulp E which happened to have an average length of 0.92 mm—equal to the number-average length of the long and short fiber mixture.

In other words, contrary to what many believed about

Table II. Effect of Mixing Long and Short Fibers of the Same Fineness

	Uniform fibers				*Mixture*
	Long C	*Medium D*	*Short E*	*Very short F*	*1/2C + 1/2F*
Weight average length, mm.	3.08	1.80	0.92	0.545	1.81
Number average length, mm.	3.08	1.80	0.92	0.545	0.92
Apparent density, g./cc.	0.513	0.508	0.513	0.518	0.535
Burst factor, metric	14.2	8.1	4.7	2.7	...
Tear factor, metric	163	71	24	11	...
Breaking length, m.	1,780	1330	1030	750	1320
Zero-span B.L., m.	10,300	9800	8300	7200	9500
TAPPI opacity,	68.1	69.1	72.2	73.5	71

Table III. Test Results on the Whole Pulp and on the Longer Fraction on Groundwoods[a]

	Pulp A		Pulp B	
	Whole	On 150 mesh	Whole	On 150 mesh
Specific volume, cc/gm.	2.2	2.6	2.2	2.5
Burst factor, metric	16.8	10.2	13.0	7.
Breaking length, m.	3300	1870	3100	1750 1
Tear factor, metric	39	42	..	...
Drainage time, sec.	51	5.5	16	6.0
Percentage of pulp tested	100	32	100	34

[a] TAPPI Standards T 205 m and T 220 m used. Fines removed by classifying and rejecting those passing a 150-mesh screen.

fiber length (including the author until this work), only the weight-average length is important; not the fiber length distribution. The latter remains important only if number averages are considered, since without stating something about fiber distribution, a number-average fiber length quoted by itself is meaningless, as we have seen.

3. The apparent density or specific volume of all the fractions C, D, E, and F was substantially the same, in other words, fiber length has no marked effect on the resultant test sheet density, which means also, contrary to what most people believe, fiber length *per se* has no appreciable effect on stock wetness. The thought that shorter fibers mean slower stock, is an old one; it is true in practice only because any method of reducing the fiber length with a refiner or beater at the same time unavoidably splits the fibers as well as cuts them, or at least concurrently produces some fibrillae and debris. It is the increased slenderness of the resulting fibers or the presence of the fine fibrills or debris, and not the cutting, that causes the shortened stock to be slower in practice.

Work reported previously, in which a pulp classifier was used to separate a normal wood pulp into fractions for the purpose of investigating the effects of length, the fractions from successive compartments used as test samples, were not only shorter but they were also much more slender. It was the increasing slenderness of the shorter fractions, not their decreased length, that made the stock wetter and gave the test sheets a higher density, a lower air permeability, and in most cases a higher tensile and burst, despite the shorter fibers.

COHESIVENESS OF FIBERS

There are no good direct ways of measuring this property, which is made up of two parts: intrinsic cohesiveness and area of contact. Probably the best measure for groundwood is the transverse tensile strength of standard test sheets, but the areas of adjacent fibers in contact, and hence the test, will be influenced by sheet density and by the fineness and shape of the fibers.

If a tensile force is applied to a strip of paper made from groundwood or an unbeaten pulp and the happenings at the line of rupture are studied, very few of the individual fibers are broken, as is the case of a strip made from a well beaten chemical pulp; the individual fibers on one side of the break are mostly pulled out from in between others on the other side. Thus the influence of cohesion is not a direct one, rather the cohesiveness of the fibers provides them with a very substantial coefficient of friction which tends to prevent them from being pulled out from between the others.

Since the burst of a test sheet increases in direct proportion to its density and also in direct proportion to the weight-average fiber length, a practical measure of cohesiveness is: Burst factor divided by the product of fiber length and sheet density.

The nature of intrinsic cohesiveness continues to be the subject of debate, although ever since the silver staining technique for making submicroscopic fibers visible was developed (*8*), it appeared to confirm the important role of fibrages, as first stated by Strachan (*9*). With groundwood, the author believes that the cohesiveness of the fibers derives from the cellulosic surfaces that are exposed by grinding and by macro-, micro-, and molecular fibrillation of the exposed cellulosic surfaces. Unlike any chemical pulp, the cellulose in groundwood contains all the original hemicelluloses and pentoses so, according to a current theory disinterred from the past and advocated by several exponents in recent years, groundwood fibers should cohere exceedingly well. It is unfortunate both for that theory and for practice that groundwood fibers do not.

GENERAL REQUIREMENTS

In accordance with the foregoing, the quest for the highest quality of groundwood pulp would appear to be primarily in the direction of more slender fibers even at the expense of some of their length. The data in both Tables III and IV would seem to confirm this because they show that it is the very fine, that is the most slender, fractions that play the predominant part in the strength of a groundwood sheet.

The results in Table III are different from those with a well beaten chemical pulp, where washing the pulp on a 150-mesh screen makes hardly any reduction to the strength factors of the test sheets made from it*; the washing merely increases the freeness to about the same level as for the unbeaten pulp (*8*).

Curves (a) in Fig. 1 illustrate the effect on tensile strength of blending groundwood fines with groundwood long fibers, both fractions being derived from their whole pulps 1, 2, 3, and 4, No. 1, being the pulp S in Table IV and No. 2, 3, and 4, groundwood made in another mill on the same grinders but under different grinding conditions. Curves (b) show the effects on the tensile strength of a mixture of the whole groundwood pulps 2, 3, and 4, with a single unbeaten sulphite. Curves (c) show the effect of the fines only from the same three pulps, blended with the same sulfite as in curves (b).

It is evident from this, that the ground wood finesplay the predominant part in the strength not only of the groundwoods and groundwood mixtures alone, but of the strengths when mixed with unbeaten sulfite. In every case the quality of the long-fibered fraction has practically no significance.

In Table IV it will be noticed that the quality of the fines as well as their quantity, apparently affect the freeness tests. This holds true in general for most groundwood pulps and since the fines are shown to be the important constituent of groundwood pulp, the use of the freeness test as a means for groundwood control has some justification. However, it should be emphasized that since fines are the unimportant

* For example, it was reported by the Division of Forest Products, C.S.I.R.O., Melbourne, Australia, at the Ninth Annual Pulp and Paper Conference held at Burnie, Tasmania in April, 1948 (Vol. 1, page 155): After washing a well beaten bleached sulpite pulp by forming sheets successively five times in a British sheet machine, the Canadian Standard freeness rose from 118 to 398 ml. but the tear factor remained unchanged and the burst and tensile strengths actually rose 2%, showing that the removal of the fines merely altered the freeness test.

Table IV. Properties of Different Proportions of Separated Long and Short Fractions of Two Groundwoods

Mixture, %		Pulp S			Pulp P		
"Short" fibers	"Long" fibers	Bulk, cc./g.	Freeness, ml. SR	Tensile, m.B.L.	Bulk, cc./g.	Freeness, ml. SR	Tensile, m.B.L.
100	0	2.4	163[a]	2970	2.4	147	2330
90	10	2.5	120	3560	2.5	162	2380
80	20	2.5	135	3200	2.9	220	1990
70	30	2.8	223	2870	3.0	260	2130
60	40	2.9	265	2710	3.2	318	2040
40	60	...	...	...	3.4	413	1750
30	70	3.3	555	2000	...	...	...
20	80	3.6	...	...	4.0	735	880
0	100	4.2	845	500	4.8	862	220
Whole pulps			243	2700	...	261	1750

[a] Short fibers are so fine that a "negative" freeness is indicated: long fibers constituted about 37% of the whole pulp separated on a Huruh's apparatus with 66 twill mesh screen.

100 g./m.^{2} sheets made on special sheet-making machine and with a lesser pressure than with the TAPPI standard method.

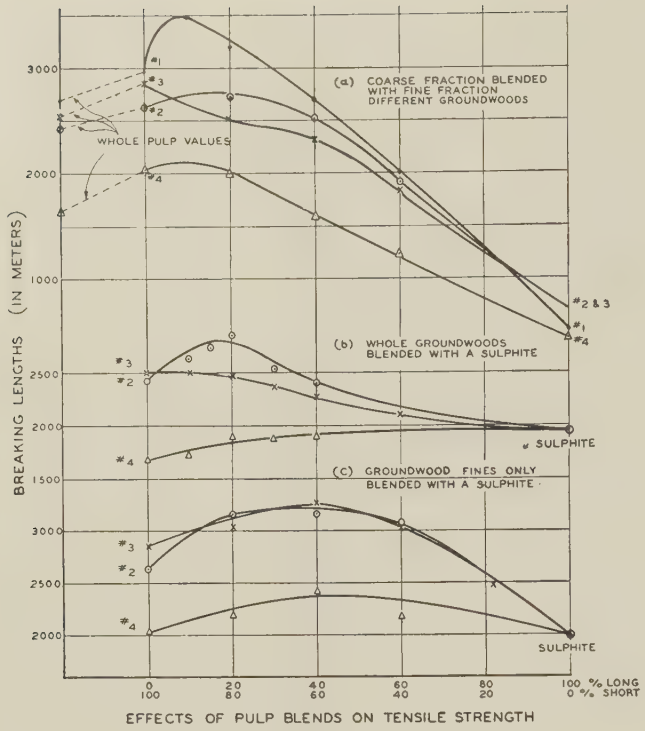
(a) COARSE FRACTION BLENDED WITH FINE FRACTION DIFFERENT GROUNDWOODS
WHOLE PULP VALUES
(b) WHOLE GROUNDWOODS BLENDED WITH A SULPHITE
SULPHITE
(c) GROUNDWOOD FINES ONLY BLENDED WITH A SULPHITE
SULPHITE
BREAKING LENGTHS (IN METERS)
100 % LONG 0 % SHORT
EFFECTS OF PULP BLENDS ON TENSILE STRENGTH

Fig. 1

constituent of chemical pulps, there is absolutely no valid rational basis for using the freeness test with chemical pulps as some original instructions for the Canadian Standard freeness test tried to make clear, and as the author has since repeatedly tried to make clearer.

It is quite evident also from Table IV that the fines from pulp S are decidedly superior. Whether their superiority resides in their greater slenderness or in their improved fibrillation is not known from the data, but because of the "negative"freeness of the entire fines fraction, it is probable that it is their slenderness that aids their quality. Also, whether it is possible to produce significantly better fibrillated fibers by grinding a given species of wood in a different way, is difficult to answer without experiments directed toward that end. In view of the good results with modern refining equipment on groundwood shives, very probably not. Better cohesiveness may well reside in a greater fiber length-diameter ratio.

Measurements of both fiber fineness and fiber length with suitable modifications of the new TAPPI Standard T 233 m and T 234 m, can be obtained in a matter of minutes—just a little longer than it takes to make a freeness test. If by some manipulation one makes a groundwood with a lower freeness, the chances are a little better than even, that the slower pulp will be superior. On the other hand if one gets a more slender pulp, or a longer pulp of the same slenderness, there can be little doubt of having effected an improvement in its quality.

It is presently not possible to contemplate how to change a given set of grinding conditions so as to get say, a higher burst, unless to remember some change that was found in the past to give the desired improvement. However, even without any practical experience, it is possible to contemplate how to get a more slender fiber which would give the required result. Perhaps as Turner (*12*) has pointed out, we might even take a leaf out of the book of modern practice in metal or glass grinding and discard the old-fashioned grindstone, employing instead an endless waterproof belt coated with a modern abrasive which possibly could be better designed to give a finer and longer pulp with a minimum input of power.

With a greater understanding of the relationship between the more fundamental properties of pulp, such as fiber fineness, fiber length, cohesiveness, flexibility when wet, and intrinsic fiber strength, all the more common properties such as wetness, burst, tear, bulk, and smoothness should be predictable. It is believed that with new testing methods, we have such an understanding within our grasp, and if so, it should enable marked advances to be made in both the quality and the production of mechanical pulps.

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Panel Discussion: Fundamental Concepts

Chairman Beasley: earlier in this meeting I introduced you to most of the head table. At this time I would like to introduce the moderator of this discussion, which will be on the fundamental concepts. I am sure there is really no need for an introduction, and that you are all familiar with the work of Mr. Gallay. I would ask that, following this discussion, any questions that are to be asked, will the questioner please state his name, company affiliation, and wait until he has the microphone, as this is being recorded. It now gives me great pleasure to introduce the moderator of this panel discussion, Mr. Gallay, research director, The E. B. Eddy Co., Hull, Quebec—Mr. Gallay.

Mr. Gallay: Thank you, Mr. Beasley. Gentlemen, I would suggest that we might relax a bit now with the lighter side of this afternoon's program. In case any of you did not know, or might have been intrigued by the question, "What sort of character called groundwood a mess at the Poland Springs meeting?" I am very pleased to reveal the answer. I plead guilty, willingly and even gladly, to being that person. At that meeting, which was held some 3 years ago and was the forerunner of the present one, I had the honor of being the keynote speaker, a term which you gentlemen from the United States will recognize from the field of politics. I had some rather strong convictions on the subject, and in developing my theme I took occasion to present those convictions. Since that time, I have had no reason to change my stand and one or two points I made then might bear repetition on this occasion.

I called groundwood a mess. I was approached subsequently by a number of people and was gratified to find that this rather drastic term had met with considerable favor and even some mild enthusiasm. The only objection which was raised was essentially to the effect that groundwood could hardly be termed a mess in view of the tremendous production of this commodity and its reasonably successful application in

paper manufacture. It need hardly be said that implied satisfaction on the score of our knowledge of a field is hardly a good departure point for further research and development in that field.

Reference was made during an earlier session of this conference to the imposing list of contributions to the literature in the field of mechanical pulp. A classification of this literature shows however that the vast majority of the papers deal with variables and interdependence of variables in the manufacture of groundwood. This is not the sort of knowledge which will lead to a new avenue of approach and a real knowledge of groundwood, but rather one only of *ad hoc* interest to groundwood operators for mild improvement in present processes. This is not to deprecate the fine efforts of the practical men in the field. Their job is one of day to-day operations, to keep up production of a standard sufficiently high to run over the paper machine with reasonable efficiency.

The fault lies rather with the programs of research in our industry, where in my view we have not seen fit to devote time and effort to a well-conceived program of basic resecarh on groundwood. Fortunately, this is not universally true, and a very few papers in the literature constitute shining examples of exceptions to the unhappy general state to which I have referred. One of the most outstanding is the classical work of Professor Walter Brecht, of the Institut für Papierfabrikation in Darmstadt. I refer particularly to the contribution which he made to the 1952 Annual Meeting of the Technical Section, CPPA, which really was the base of departure for the two papers we heard this afternoon. I commend this particular paper of his to your thorough study, if you have not already done so.

For many years, various workers have devoted a great deal of attention to the "mechanism of the grinding process," i.e. the comminution of wood by the grindstone. This has had the avowed goal of reduction in power expenditures mainly, with some additional interest in variations in freeness, etc. We are pleased to speak of groundwood fibers and tend to forget at times that only rarely do we obtain fibers. We produce entities which are actually bits and pieces of wood, and we have confused the issue by attempting to characterize them in the same fashion as we employ for true fibers of relatively uniform cross section. The evaluation of these various entitites which constitute groundwood has received altogether too little attention. It appears to me to be illogical to attempt to improve a material when we have little knowledge of what we want in its constituents. Therefore, in my view we must study the evaluation of groundwood before attempting to elucidate a particular mechanism of grinding. It seems altogether strange that we should attempt to effect improvements when we know little of what we are trying to accomplish.

In view of this, I personally am very gratified that the mechanical pulping committees of the Canadian Technical Section and of TAPPI have devoted a whole session to this topic of fundamental concepts of groundwood evaluation. I am convinced that it is the most important topic in this whole field.

Let us proceed to the discussion of the afternoon. We have a rather unusual panel; a group of individualists might be a better term. For example, I very seldom agree with my friend Jim Clark. Jim Clark and Boyd Campbell seldom agree with each other or, for that matter, with almost anyone else. Borje Steenberg has firm views on nearly all phases of pulp and paper and he has an uncanny ability of backing up those opinions with magnificent logic and reasoning and even rhetoric on occasion. Christian Anker is of course an elder statesman in the field of groundwood. Allan Robertson is well known to us from his published researches and we heard from Mr. Herwig earlier this session. In preparing for this session in what seemed to be an orderly fashion, I wrote to each of these gentlemen suggesting individual assignments for which they might make themselves responsible. I met with a rather lukewarm response. At a breakfast meeting this morning, after a further organizational attempt had met with equally little success, it was settled that we would simply agree to disagree if the occasion warranted. We shall therefore start without bias, it is hoped, and certainly without specific organization.

In order to avoid complete confusion, however, I suggest that we discuss phases in some semblance of order. Let us assume that our groundwood has been made and that shall follow its course on the paper machine and its application subsequently. We might take a glance at what is involved in getting rid of water on the wire and then examine some properties of the wet web at the couch and in the press section. Then we shall talk about some properties of the dry sheet and eventually include something of specific applications. I hope that our audience will participate in the discussion with the panel.

I am going to ask Mr. Campbell to lead off with any comments he has—Mr. Campbell.

W. B. Campbell: We are all supposed to be experts up here but I don't know very much about drainage. I have been interested in it for about 30 years, from the point of view of freeness which Jim Clark curses. I became interested trying to make the freeness tester more usable. I was never, at any time, under the misapprehension that freeness told the whole story about groundwood or about any other pulp. Actually, if you look back at the history of how the freeness tests originated you can get some notion of what it is all about. The freeness test originated probably before there was any such thing as groundwood. The old beater man used to (and beater men still do, where there are beaters), stick his paw in and grab up a handful of pulp. He feels it and squeezes it and sees how the water squeezes out, and, maybe, puts some on a blue glass and looks at it, but a great part of what he finds is in the feel of it, its slipperiness and, particularly, how the water squeezes out. That tells him a whole lot about it. Some people, starting from that point, said, "Let's measure how tight you have to squeeze, or how this loosing of the water is affected. Can we measure that?" Well, the Schopper-Riegler was one of the early testers, and the Canadian freeness was a modification of it. Freeness testers, all of them, measure essentially, total surface, but not exactly because the drainage depends on how tightly the pulp packs in the freeness tester, as well as on the total pulp surface. So that you have a mixture of two different factors there. Unfortunately, as shown by some of Mr. Robertson's work, while any one kind of pulp will give you nearly a straight line of freeness against total surface, the straight line for one pulp does not agree at all with the straight line for another pulp. The slope of the line is just about twice as great for sulfite as it is for kraft and considerably more than it is for groundwood. That is a confusing factor. When it comes to drainage on the paper machine, and that is, apparently, what Gallay wants me to talk about, I don't know very much about that, in fact almost nothing. Thinking of newsprint particularly, you put on the machine a lot of pulp of which about half disappears through the wire. You are only operating on about half of the pulp that goes on to the furnish of the machine. The other half that goes through the wires, you catch and put back in. It is extra, and it goes round and round and gets in the way. But when it comes to draining through the wire some of that pulp that drains away is just like so much water; it interferes a little bit, but not very much, and the net result is that what stays on the wire and really affects the drainage is pretty near the furnish that stays on the machine.

Then you have a factor of compressibility. It has been shown that the pressure (or the suction, which comes to the the same thing), on the wire of the paper machine is related to the speed of the machine. Actually, it is very nearly a suction equal to the velocity head corresponding to the

speed of the wire. So, therefore, the compressibility of the pulp must enter into the drainage rate. The compressibility, as Jim Clark says, is a matter of the fineness of the pulp. Robertson's work shows that there is a factor which, at first sight, seems quite contradictory. Whether pulp is ground or is beaten, the process is in principle; the same. They are both functions of breaking bonds in order to reform them on the paper machine, but Robertson's work shows that in the beating and in the grinding there is a swelling of the fibers, and by fiber I don't mean only the fiber as it is in the wood or the fiber that is in the pulp as fibers, but all the material. The specific volume in water of the beaten stock is considerably greater than the specific volume of cellulose. In chemical pulps the swollen volume amounts to three or four times normal volume, in groundwood about half of that. If you allow for the fact that groundwood is only 50% cellulose, the dilution by swelling is about the same as it is in the chemical pulp. Robertson has shown that the swelling, the increase in specific volume, comes about very early in the beating. I don't think you can show that with groundwood because you don't have a chance to get at it in quite the same way. Doughty's work showed that if you beat pulp a little bit, and then get a dense sheet by compression afterwards, you have very nearly the same effect as if you had got that same sheet density by pure beating a longer time. However, Robertson's work shows that the strength of the sheet follows the same curve as the specific volume if you plot is against total surface. Jim Clark says you have got to have it fined. Now, some of that fineness may be the fineness due to internal fibrillation which makes for that swelling. That is probably why the swelling takes place. So, while they are contradictory in one way, they are not in another. In either case you get something that can make for the greater total surface.

Mr. Gallay: Thanks very much, Boyd. I think we might profitably pursue this matter of freeness just a little further. Dr. Clark, in his paper, showed a great scatter sort of a diagram, when it was attempted to relate freeness to productivity off the machine. I am suggesting—and I am going to ask for comment on this—I am suggesting that that is because the same freeness can be obtained by an infinity of routes. You can have all kinds of mixtures of these groundwood fibers and a great many of them, quite different groundwoods, will arrive at the same freeness because of the nature of the freeness test. What about that, Jim?

Mr. Clark: Yes, I think that is very true. That is why I don't think freeness is any good because you don't know of what it is a measure. Freeness is an over-all measurement of many, many things, mostly debris, also flexibility, as my friend Boyd just pointed out, and fineness as you pointed out. It is so complex that it is impossible to analyze it. When we first developed the Britist sheet machine, I believed in the freeness-burst-gear system too. But it failed me so often that I had to give it up. I am trying to resolve this most complex—pulp quality—into more simple parts, and look at it under simple factors: length, intrinsic strength, wet flexibility, and cohesiveness. These are measurable factors, and I think with them firmly in mind we can then get away from the maze of complexities that come with drainage "one" and drainage "two" [the upsetting results of using] different freeness testers and the concepts of "hydration," and so on. The old system is so darned complicated that I really think that perhaps only by approaching it from the more simple way I have suggested, can we get a picture in our minds that is clear enough to use to make progress in both the design of grinders and the improvement of the pulp.

Mr. Gallay: It should, however, be emphasized that the concept of freeness has been a very useful one to the industry, despite its limitations and particularly despite the misuse which has been made of it. If a groundwood mill is run in much the same way from day to day and the product varies within normal limits, the measurement of freeness provides useful results. It seems to me to be obvious that a comparison of machine productivity over a variety of mills all over the continent on the basis of groundwood freeness is bound to be confusing and even misleading. I wonder whether Mr. Robertson would like to add to this discussion of drainage.

Mr. Robertson: You have put me in a rather awkward position here. I am gratified at the faith that Mr. Campbell has put in the measurement of specific surface and specific volume, but I am afraid that I will have to disagree with him about the particular usefulness that this has to groundwood tests. The measurement of swelling and surface is valuable in the evaluation of chemical pulps. With groundwood pulps the significance is very much less. I have become a disciple of Dr. Klemm's and Dr. Brecht's who take care to distinguish the effects of flour stuff and slime stiff among the fines. Both of these have large specific surfaces. The simple measurement in specific surface does not separate these things although flour stuff is bad fines and slime stuff is good fines and the significance is limited. As far as the measurement of swelling is concerned, I think it has limited significance here too, particularly in relation to the larger fibers. When the wood is ground you still, essentially, have the wood structure in the larger particles and I think this shows relatively little swelling or collapse. You have rather stiff particles. They have not the freedom to collapse and they do not have the freedom to swell. In chemical pulps this freedom exists; the fiber can collapse, the fiber can swell. In groundwood particles this is not the case except, perhaps, in the slime stuff. So, although I am associated with the method I feel that I must, in all honesty, express my doubts as to its validity in groundwood evaluation if it is applied to the whole pulp. I make this statement in the belief that the proportions and quality of the various groundwood components must be determined to evaluate the pulp.

Mr. Steenberg: Well, I think we have to remember that the process according to which we make groundwood is a random process, and in such a random process as trying to disintegrate a fibrous material we can, in all probability, never hope to get a tree from chipped-off material from these fibers. If you try to shorten a classical wooden pencil you know how very difficult it is to have all these wood chips stick to the pencil, I mean, if you start to shave them off, simply because the main work is the one to start to peel off one of these chips. Once it has started to peel off there is very little energy to take it off completely. Take a piece of scotch tape put onto some surface, it is difficult to start it, but once you have got the angle, practically no energy is needed. So, in that sort of shear process which we have in groundwood production and which we, of course, also have, when we beat chemical pulp, we are bound to get chips, and these chips are, in good mechanical pulp, the main interesting thing for the freeness. In poor groundwood we will get drainage resistance essentially from flour-like products. So this means that you can have the same freeness, in one case from dry wood flour, in the other case from the long shavings. Actually, they are possible to see in the microscope. Rather few people have looked at it because you can hardly see them in normal microscopic technique due simply to the fact that you don't look for them and you use, generally, coloring techniques and the dyestuffs you use for coloring them dissolve or soil them so deeply that you don't see them. If you take a face contrast microscope and look at it without any coloring you would be amazed to find curly, horsehair-looking material all over in all good sorts of groundwood, and this very fine material is the material which is what is called schline stock or schline material by Prof. Brecht because such a curly, fine material keeps water and, on drying, it shrinks which, of course, the flour doesn't. Now, the presence of such curly hair is of no interest, as a matter of fact we are not interested in it at all from the drainage point

of view, but it will always follow the production of long, slender fibers because in taking out a long, slender fiber that has to have a fibrillar structure, you cannot take it out alone. It must, because of the randomness of the process, chip up a part of it and, consequently, you will always get a certain relation between freeness and the properties of the pulp within some reasonable reason. If the pulp is good the drainage resistance is due considerably to this fine haired material, and that is the explanation for the fact that so many groundwood people, I would say practically everybody, sticks to the freeness test as a sensible one. It measures something that, for fairly good groundwood produced, is parallel with existenence of slender fibers, and it also is parallel with this slimy, hairy material which can, due to its slenderness, shrink and fill up the structure, giving strength while flour just fills it out, without giving strength.

Mr. Gallay: Thank you, Professor Steenberg. Earlier this afternoon, Mr. Herwig made detailed reference to the theory of Professor Brecht on drainage. This involves in part essentially opposed requirements in the table roll and vacuum box sections, insofar as drainage properties are concerned. There has been some intimation of disagreement with this view. I wonder whether you would care to enlarge on this, Mr. Herwig, from the point of view of actual findings in large-scale practice, and in particular whether it is necessary to contemplate a compromise in the properties of groundwood in order to obtain optimum results.

Mr. Herwig: I think I mentioned the essential features of drainage effect "one" and drainage effect "two," but in order to clarify that a little bit more I would like to talk out of practical experience. We used to have groundwood pulps ranging in the freeness of 80 to 90 which would correspond to a drainage effect "one" of around 1.8 to 1.9. We observed a vacuum build-up in our suction box and suction couch. In the suction box it was around 8 to 9 in. and in the suction couch around 15 to 16 in. After about half a year we slowly decreased the freeness. We experienced a drop of around 0.5 and the drainage effect went down to around 1.4 to 1.5. The freeness went down to around 40 to 45 and 50. At that lower freeness and at that lower drainage effect "one," at the same basis weights of the paper produced there were no changes in the suction boxes or the suction couchs. Drainage capacity maintained or the pump capacity maintained at the suction boxes and the suction couch—the increase of the vacuum in the suction box was around 12 to 13 in. and in the suction couch, 21, 22 and 23 in. was the general range. Moisture tests at the wet end of the paper machine—between the couch and the first press, at the higher freeness and the higher drainage effect "one," we had a moisture content around 17 to 18% dry, of the sheet leaving the couch. After decreasing freeness in drainage effect "one" to a low level, we had over-dry content of the sheet at the couch of about 19.5, 20, and 20.5%. Now this was definitely before we knocked down the drainage at the table roll part. We closed up the sheet at the suction boxes or the suction couch would be fed through the higher density of the sheet. We obtained a higher speed of the air penetrating the sheet into the suction box and a higher velocity with higher secondary drainage at the couch. We went so far that we had the drainage effect "one" down to around 1.1, drainage effect "two" up to 2.5, and at this time the drainage characteristics reversed, for we went into the suction box and suction couch with a rather wet sheet. Due to the limitations of the vacuum pumps we just couldn't draw enough air through the sheet and we had a lower over-dry content of the sheet after the couch. So, what we finally did, we increased the drainage capacity of the machine by putting in a wider suction box and then a new vacuum pump of higher capacity. From this time on we ran that machine at 1045 f.p.m. Before, we couldn't get it above 960 to 970 f.p.m.

Mr. Gallay: Thank you very much, Mr. Herwig. I would interpret those remarks as meaning that there was an excellent confirmation of the Brecht theory of drainage in various parts of the wire. Mr. Campbell, did you want a chance at rebuttal of some of the remarks made on drainage after you got through?

Mr. Campbell: Now, Jim Clark has damned freeness, because it doesn't measure any one thing. Now, I think that is an improper way of approaching it. Papermaking is an art. I don't believe, at any time, even 50 years from now, we will be able to get a number of factors which, put together, will describe the groundwood that is being made. I have, over the years, acquired a tremendous respect for the art of people dealing with any one material day after day. I think that our groundwood people know a whole lot more about groundwood than they are able to express, or ever will be able to express. Freeness tells them some things. The blue glass tells them some other things, and by a sort of intuition they can relate one to the other and, probably, include a whole lot of things that they don't conciously realize. It is something like this. We all recognize a pretty face. But if we started to measure the length of nose and compare it with the length of nose of somebody else we would have some numbers, but the numbers themselves wouldn't mean very much. But we can look at faces and have a pretty darned good sense of what is a good-looking girl. And I think that papermakers can have a pretty good sense of what is good groundwood if they know the freeness and have had experience in looking at the pulp under the blue glass. Robertson has sort of sold off some of his work, I think, unduly. He says that he doesn't think that the groundwood swells very much but I don't think that he has done enough calculating on that. His own figures show that the specific volume of the groundwood is something more than twice the volume of the wood cells including the lumen. You can calculate back from the specific volume of the wet wood or dry wood and compare it with his figures for specific volume, and his figure works out to be about double the specific volume. So I think there is something to be said for the plain groundwood man that really looks at things.

Mr. Gallay: Thank you. It is encouraging to learn that data on the swelling of groundwood may be of interest in connection with groundwood in parallel with a similar relationship in the case of chemical pulps.

Prof. Brecht: I would like to add a few comments to this interesting discussion on drainage. We have continued our earlier studies, referred to several times during this meeting, particularly with a view to reducing complexities to the ultimate. For efficient use on a high-speed news machine, the groundwood must have a high proportion of long, slender, flexible fibers. This, however, is quite insufficient for characterization. There must also be included the correct quality of fines, i.e. slime material, and the importance of this fraction can hardly be overemphasized.

In the meantime, we have developed what would appear to be a very simple method for the evaluation of drainage characteristics. This involves essentially the measurement of the air porosity of the wet web. We have found that the specific surface of the slime material is many-fold that of the groundwood as a whole, and is also far greater than that of the flour material. It would appear from our work thus far that the air porosity of the wet web will reflect essentially a composite of all the factors which have been mentioned in this discussion. In addition to the proportion of slime material, the compressibility of the wet web is also included in such a measurement. This very simple procedure obviously offers a very rapid test for the quality of groundwood. This evaluation has already had an instance of verification on a commercial scale. In this case, it was shown that we could extend the evaluation to the screen room and we were in fact able to predict within an accuracy of $\pm 10\%$ the power consumption during screening. In this work, the governing factors again were the proportions

of long, slender, flexible fibers and of correct quality of slime material.

Mr. Gallay: Thank you, Professor Brecht. I have one query on your very intriguing concept of the use of air porosity for pulp evaluation. I presume that this test would be very sensitive to the moisture content of the wet web just as in the case of wet web strength and stretch.

Prof Brecht: Yes, it certainly is. It is essential to measure the moisture content in addition to the wet web strength.

Mr. Clark: Would you not agree with me that air porosity of the wet sheet or rather a damp pad of pulp, would primarily be a measurement of two factors. The amount of fines, perhaps, is the minor one, and the major one, the wet flexibility of those fibers or, as we have tried to point out today, their slenderness.

Prof. Brecht: That is quite true, and I have not been able to separate these two factors and make a distinction between them.

Mr. Clark: Well, you can, of course, on a classifier, measure the amount of fines separately. Would you not, also, agree with me that the drainage "one" is almost, primarily, a function of the amount of debris in the pulp? Because, if you have a pulp and put in 2 or 3 tablespoons of white water drainage "one" goes away up; it takes much longer to drain. All you are doing is adding a little more fine flour. From many experiments it seems clear that the drainage time of making a sheet almost wholly depends on the fine material or debris present and then, I think, the drainage "two" that we were talking about, depends partly on the amount of this fine material but still more on the very important factor of slenderness of the fibers which causes a more or less densely packed pad.

Prof. Brecht: I agree, but I have no proof for the first point which you made. The measurements which we made involved the method which Mr. Herwig has described, but we had accompanying data on a paper machine. Since the latter was relatively slow, we were able to determine the solids content of the web just prior to the first suction box. There was a definite correlation between this solids content and the data we obtained from my Fig. Al, to which you referred. I have no information, however, on the relative roles played by these two individual factors.

Mr. Gallay: While Prof. Brecht has the microphone, would anyone like to question him on any of these points? Mr. Atack, would you like to ask a question of Professor Brecht?

Mr. Atack: I don't think that we should overlook the fact that it is just as necessary to qualify the term groundwood "fines" as it is to qualify the term groundwood "fibers." The very beautiful electron micrographs of beaten chemical pulps which have been obtained by H. W. Emerton and his colleagues indicate that some of the fine material is not fibrillar in nature at all, but is in the form of membranes (Emerton, H. W. Watts, J., Amboss, K., and Simpson, A., B.P. & B.M.A. Report RA/T/38, July, 1954). This is supported by similar studies on the phase contrast microscope (Van Nederveen, G., and Isings, J., *Tappi* 37, No. 3:103 (1954)).

Since the last International Mechanical Pulping Conference at Poland Springs we have had an opportunity to examine samples of groundwood fines, which contained predominantly "flour stuff" and "slime stuff," on the scanning electron microscope. These samples were kindly provided by K. H. Klemm. Several impartial observers attempted to pick out any significant differences in geometry between the two samples without success. Since these samples were freeze dried it was a little surprising to learn from Professor Steenberg that such differences may easily be detected in the phase contrast microscope.

My final point is directed to Professor Brecht and Mr. Robertson and deals with the measurement and significance of the surface area of groundwood fines. I wish to ask Professor Brecht whether he attaches any significance to the differences in surface area of the "slime" and "flour" fractions he obtained using the permeability method. I gather that these measurements are particularly difficult to make on fines samples in view of the plugging which occurs. Moreover, the results may be a little difficult to interpret. Mr. Robertson has stated that in his opinion surface area measurements on groundwood fines are of little importance. Perhaps he would care to qualify this statement.

Mr. Steenberg: To Mr. Atack I would like to say that I understand his point of view. When I first read the Brecht paper on "slime stuff" I had a very strong feeling of wishful thinking, partly because Brecht was not able to show a photograph of the material but only a drawing and partly because he attributed such great importance to a small fraction of the stock, a fraction so small that it was overlooked by others. I have changed my mind. Today I know that it is practically impossible to see this material in an ordinary microscope, while it is easily seen in a phase-contrast microscope. It changes its appearance considerably on drying and consequently would look different in an electron microscope, where it is studied under vacuum. With a new type of fractionation device in my laboratory we can separate this material on a large scale, not only from groundwood but also from beaten chemical pulps. We can do it on pound scale and at least as far as chemical pulps are concerned, the material is practically completely free from fibers or fragments. I will be happy to send you a batch and you will find that it contains thin membranes and fibrillar material. Electron microscope investigations indicate that this curly fibrillar material may originate from all parts of the cell wall. When fibrous material like wood or pulp is subjected to forces which may cause fibrillation it is very improbable already from pure chance considerations that the peeling-off action will stop before the fibril has become completely removed. When fibers appear fibrillated it is consequently necessary that in the stuff should be present a much larger amount of free fibrills than those seen attached to the fibers. It is remarkable that this point is generally not stressed in papers dealing with fibrillation of fibers.

Mr. Gallay: Now, what about the second question? Professor Brecht, would you like to add anything to this statement about the interference in that surface area measurement from the point of view of plugging, using the Mason-Robertson technique?

Prof. Brecht: We had hoped that by measuring the specific surface of the whole groundwood and also the specific surface of the fibrous portion, i.e., that part retained on 200-mesh, the difference between these might give the specific surface of the slime material portion of the fines. We did not succeed in this endeavor. The reason I believe lies in the method used for measurement of these specific surfaces. Such a method simply cannot be used for both a very wet or slow material such as the slime and still be useful for the flour material. This is simply not possible. The same form figure in the equation cannot be used for such highly different stocks and this I believe accounts for the lack of success we experienced in our work. For this reason, we are now trying out air porosity of the sheet instead.

Mr. Gallay: Thank you very much Prof. Brecht. Before coming to the third question asked of Mr. Robertson, I would like to make one remark. In connection with nomenclature, I objected to the term "fibers," and Mr. Atack was quite right in that this must be extended to fines also. One matter has concerned me in this connection. Flour material belongs in the fines grouping; it is not a fiber by the ordinary definition of a fiber. A fiber depicts a material whose length is much greater than its cross section. The flour would appear to be bits of entangled debris. In the case of the slime stuff, where does one draw the line between fines and fiber? Why pick on 200-mesh or any other specific figure? At what point in size do we draw a distinction between a big boy and

a little boy? I presume that we actually have the complete range of lengths from the whole fiber down to the slime. Particularly in view of the fact that various people do not even pick the same artibrary cut-off point I, wish to emphasize the possible loss of perspective involved in these distinctions. Now would you like to continue, Mr. Robertson, with your answer to the third question from Mr. Atack?

MR. ROBERTSON: My argument about the measurement of specific surface of the pulp not being important because it couldn't distinguish flour stuff from slime stuff was simply make on the basis of the consideration that these two substances are finely divided and logically, therefore, they both have a very large specific surface. We haven't gotten as far as Professor Brecht in separating the two and trying to measure them. The thought was simply that they are both fine materials and the method of measuring the surface area of the whole pulp does not distinguish them.

MR. GALLAY: Thank you. Mr. Clark would like to have another word on the subject.

MR. CLARK: I would like to comment on this difficulty that has already been mentioned, about the difficulty of getting an appropriate constant when you are measuring the specific surface in accordance with the porosity method using the Kozeny equation. I wonder if we shouldn't just realize that a result may be a mathematical figment, in some instances, unless we know the proper constant to use. But the point I would like to make is this. Why get things so complicated with terms like "flour" and "fine stuff," in either German or in English when, after all, what we are talking about is only differences in the ratio of length to diameter. My plea is, don't let us talk about such difficult things. The "slime stuff" is merely hairlike fines, long fines, and the "flour" is short fines. So if we know the ratio of the length to diameter, on a weighted average basis I think we can simplify the matter.

MR. GALLAY: Professor Brecht pointed out in his major paper in 1952, if I remember correctly, that the axis ratio was actually the major item of difference between flour material and slime material. Is that correct?

PROF. BRECHT: I regret that I cannot agree with Mr. Clark. I believe that there is a profound difference in the properties of these two components. The fines, in my view, are not simply composed of flour material and slime material with gradations from one to the other. The properties conferred on the sheet by these two form components are, as we have shown, entirely different. They can be separated from each other readily by sedimentation methods. The addition of the flour material to a stock results in a degradation of all strength properties. On the other hand, the judicious addition of slime material enhances strength properties handsomely. It is obvious then why we are convinced that we must have a means of measurement of these two form components separately. If these two components differed only in dimensions, we would have little or no interest in distinguishing between them.

MR. CLARK: But I don't think we disagree at all. I agree with your statement of the effects. All I am asking is that you agree with me that the "flour stuff" is short, fine material, the "slime stuff" is a long fine material, and we can explain the differences. The differences are profound I agree with you, but they reside in the differences in dimensions of those elementary particles.

PROF. BRECHT: I do not agree. I believe that the swelling properties of these two components are quite different, as are the general colloidal properties.

MR. CLARK: They are both all wood substance.

PROF. BRECHT: Dimensional differences form only a small part of the consideration.

MR. CLARK: Well, what else is there? They are wood

PROF. BRECHT: Obviously. But consider the difference in the following light. In order to prepare quantities of flour material for investigation, we had to use dry wood and very intensive high pressure grinding. The dust thus produced, when further comminuted, resembled flour material closely. On the other hand, for the preparation of large proportions of slime material, we had to start with very moist wood and carry out our grinding at low speed under low pressure. On removal of long fibers from this stock, we obtained an entirely different type of material to the previous flour. We produced slime material with vastly different properties. Perhaps, I do not understand the point you are making.

MR. CLARK: No. That is the point. I maintain that they are the same substance and it is merely a matter of their dimensions, and you say they are different; we have an honest point of disagreement there. Some day, perhaps with an electron microscope, this matter can be resolved. I say they are both wood. One is long and fine and the other is short and fine.

MR. GALLAY: Thank you, Professor Brecht. Other factors should be considered aside from size and distribution of fiber entities. The chemical effect of heat produced during grinding on lignin and other organic constituents of the wood might produce varying results among these fractions, leading to differences in the surface properties discussed by Professor Brecht. These are important matters in addition to purely dimensional considerations.

MAUNG HTOO: I was thinking one of the things that could enter into this hydrogen bonding is one thing. It could be wood. But you could have various degrees of hydrogen bonding with different fibers, I believe.

MR. GALLAY: Well, I propose that we talk about hydrogen bonding under another topic in a few moments. Would you hold your question until then?

MR. CAMPBELL: I would like to point out to Jim Clark that there is no such thing as just wood, even in the single fiber. There is considerable difference in the amount of lignin in the different layers of the fiber and, as regards what Professor Brett says, I think he is quite right. When you grind up the dry wood you do get little chunks of the original fibers, because those little chunks are held together by the original hydrogen bonding, which we are going to talk about sometime later. But when you grind it under gentle conditions it corresponds to what, in beating, is termed "wet" beating, where you tease out the actual fibrils of the fibrous structure, and that teased-out fibril mass constitutes the "slime stuff" of Mr. Brecht's, and that is the stuff that is pretty effective in bonding the sheet together when you actually make the paper of it.

MR. GALLAY: Now we appear to have progressed to a wet web containing about 20% solids. We are intensely interested in the strength and stretch of the web at this stage, and as a matter of fact this interest retains considerable magnitude even at the much higher solids contents in the press section. In my own laboratory, several years ago, we investigated the physical properties of webs containing as low as 7% solids, i.e., corresponding to a web well back on the wire. Since that time, Mason and Robertson have investigated the situation at still much lower solids contents. We had speculated that some residium of this structure should be retained even in fiber suspensions. I would like to ask Mr. Robertson to comment on his work.

MR. ROBERTSON: Mr. Herwig has already introduced the subject of wet web strength and its importance is obvious. needn't elaborate on that. His paper has shown a very remarkable intercorrelation between drainage and wet strength. I think, however, that we probably have to look at the problem of wet strength a little more fundamentally. Mr. Herwig's method would be, presumably, an excellent control method but when we come to evaluating groundwoods which are made by different processes, for example groundwoods based upon refiners or groundwoods based upon such techniques as Mr. Bersano described this morning, or when we are evaluating different wood species, we need a little more information as to how this wet strength originates and de-

velops. May I remind you that the structure of the web at the couch is a three-phase system. It consists of fiber, water, and air, and the strength of this web depends upon several factors. One is fiber entanglement; another is the surface tension of the liquid; another is the interfiber friction forces, the frictional resistance to sliding of fibers over one another and, finally, bulk and per cent solids content. The bulk and per cent solids content, of course, are simply measures, in another way, of the fiber-water-air ratio. The latter two, bulk and per cent solids content, depend upon the drainage characteristics as Mr. Herwig has pointed out and upon the compressibility and springback of the web on the wire due to the drainage forces involved. Mr. Herwig has produced high significance limits for the relationship of his drainage properties with wet web strength and with the efficiency of the paper machine. If, by any chance, these things should fall below the 99% level, one might look back at the other elements involved, not bulk and not per cent dry content, but fiber entanglement and the interfiber friction forces, and for this purpose I would like to present a small test which was devised in another connection, but which might be useful here in interpreting the effects of fiber entanglement and interfiber friction forces as they affect wet-web strength. The test I am referring to is the network strength test, and the apparatus is indicated in the first slide. We have, on the right hand side of the diagram, a very simple small circulating system which consists of a vessel containing a quantity of fibers, and these fibers are circulated, by means of the pump shown, through a piece of rubber tubing and a glass tube. The dimensions of the glass tube are something between $^1/_2$ and $^3/_4$ in. diam. and, perhaps, 15 to 20 in. in length. This pulp is circulated as rapidly as possible by the pump without entraining air and then the circulation is abruptly stopped by pinching off the rubber tubing. Thus a highly dispersed pulp suspension is suddenly arrested in the system, and in our glass tube we have a uniform distribution of fibers. We then remove this test tube from the circulation system and we suspend it in the top of another tube, as shown in diagram B, which is somewhat larger and is filled with water. The level of the pulp suspension is allowed to fall to the level of the water in the large outside tube, and this initial condition having been realized, we allow water to drain at a constant rate from the whole apparatus by moving down a constant level device. That is, water is drawn from the large outside tube and the pulp suspension from the inside tube flows out into the surrounding water at the same velocity at which the water is moving. Under these conditions, and at consistencies anywhere from 0.3% up, this pulp which flows out will form a pendant flock at the end of the tube and as the drainage continues, the pendant flock will grow in length until it can no longer support its own buoyed weight and the flock will break off and fall to the bottom of the tube. The measurement of the breaking length of the flock is a measure of the entanglement of the fibers and the interfiber frictional forces, quite apart from any drainage characteristics or any surface tension effects. The second slide shows some photographs which we have of the flock forming at the end of the tube and the flock breaking off. The outside tube is shown with the inner tube suspended in it; the water and the flock are moving down because we are draining water from the bottom of the apparatus, the pendant flock grows in length and finally breaks off. Now this, of course, is a test which evaluates several things in combination. It evaluates fiber fineness, fiber length, wet flexibility, and frictional interfiber forces. I offer this test although we have not yet proved it to be of any value as a control method, because we don't have any facilities for evaluating running quality of pulps. At least it is a corrolary of Herwig's and Brecht's method in that it looks at different factors affecting wet web strength and at most, since all these factors seem to be interrelated, it might possibly, if someone would prove it for me, be a control method of pulp quality.

Mr. Gallay: Thank you, Mr. Robertson. This ingenious technique is an intriguing one, and will undoubtedly be of great assistance in filling in an important gap in the whole spectrum of strength development in paper. I would however like to make one specific point in this connection. It seems to me to be of considerable importance to include a surface effect at very low solids contents, which may play an important part in wet web strengths and in flocculation of suspensions, as well as offering a continuity leading eventually to bonding.

Mr. Clark: You are talking about fibrillation?

Mr. Gallay: No, no, definitely not fibrillation! I am speaking about surface characteristics which, according to our present concept of interfiber bonding, must be available. It must be borne in mind that we do not change the surface of the fiber on the paper machine as we remove water and attain a higher and higher concentration of solids. Whatever chemical and physical properties we have available for bonding are there in the early stages on the paper machine, and it appears to me to be entirely logical to regard these characteristics as capable of exerting very appreciable effects even where the pulp consistency is very low.

Question: Are you talking about interfacial tension?

Mr. Gallay: No, I am not talking about interfacial tension as such. Let me put it this way. I assume we are all in agreement that for eventual bonding during the later stages of drying, we require exposed hydroxyl groups which will be brought into sufficiently close juxtaposition to effect interfiber bonding on drying. These hydrophilic centers must also be available in a suspension having 0.5% concentration of pulp, or at any other concentration. What I am strongly suggesting is that we take into account the possible interplay between such groupings when brought into mild contact without the strong forces eventually exerted by surface tension during actual bonding. This type of adhesion resulting from an appreciable amount of what may be termed tackiness on the fiber surfaces will contribute materially to the strength of wet webs. The same sort of reasoning is applicable to flocculation as well where, in addition to purely physical entanglement, an interfiber adhesion would add materially to the tendency to flocculate at low suspension concentrations.

This whole subject of wet web strength is open generally for discussion.

Mr. Mayer (Oxford Paper Co.): I would like to clear up a point regarding Mr. Herwig's work. He employed Becht's original wet web strength evaluation technique. Brecht formed handsheets on a Rapid-Koethen sheet mold and subjected them to a fixed drainage schedule by means of a positive displacement sump pump. The moisture content of the wet pad was assumed to be an index to the drainage characteristics of the pulp. The wet web strength of the specimen was determined at the moisture content produced by the apparatus. The strength of course varies significantly with the moisture content.

Brecht's technique, however, resulted in wet handsheets whose solids content increased as the freeness of the pulp was decreased. That result is more or less contrary to intuitive thinking and contrary to papermaking experience.

Later on a Scandinavian investigator, Nordman, used a traveling suction box underneath his sheet mold wire. His results contradicted Brecht's, that is, he obtained lowest solids with slow stock. This is highly significant to me, because the traveling suction box device should be more related to a paper machine than a stationary sheet mold. Brecht may have obtained low solids content for the free sheets, because the droplets of water hanging underneath the septum were sucked back into the sheet thereby increasing the moisture content of the sample. Nordman's apparatus probably doctored those droplets off from underneath the wire.

Dr. Gallay: Professor Brecht, would you like to answer that?

Prof. Brecht: I can answer that directly. Mr. Nordman was kind enough to give me permission to build his apparatus. He is quite right and we were wrong in our earlier work. His apparatus and the results obtained with it are much more closely related to the conditions on the paper machine than the method we had used previously. I have no doubt on this point. We published this about a year ago and gave our findings and interpretations in detail. Mr. Nordman has done very fine work in developing his excellent apparatus.

Mr. Mayer: That brings up the item: how did Mr. Herwig do his work? Did you follow the original Brecht technique throughout? That is, did you allow the sheet moisture content to level off at the natural value just using the suction pump of the rapid Koethen sheet mold?

Mr. Herwig: We did not doctor the water off.

Mr. Mayer: I would also like to know if you used the natural moisture contents when performing the wet strength test.

Mr. Herwig: Yes, I did.

Mr. Mayer: That puts a little different complexion on your results as far as I am concerned. I am very interested in this and I am glad that you obtained a good correlation between your wet web strength rests and paper machine efficiency but I will be suspicious of the results as long as 10-day moving averages are involved. Another subject I would like to bring up. We have here been complaining about the Canadian Standard freeness tester, how insensitive it is, how freeness data fail to correlate with operating experience, etc. I believe most of us will agree that it is quite useful when employed on a given pulp or a furnish of relatively fixed composition. It is well known that it lacks sensitivity in the low freeness range (below 100 ml.) and that is because it will give readings approaching zero. A zero "freeness" is a meaningless figure. We have, by the way, obtained zero freeness pulps in the laboratory. On the other hand, the instrument is hypersensitive, numerically, in the free range.

We have obtained excellent correlations between the freeness and Gartshore drainage time or TAPPI drainage time of a given type of pulp refined to various freeness levels. We have also obtained very good correlations between groundwoods of various freenesses and their specific filtration resistance, as determined by an apparatus modeled after the one at The Institute of Paper Chemistry. So the Canadian freeness tester is not completely worthless.

For those who do not like the Canadian freeness tester I would like to point out that a suitable substitute is available, the TAPPI sheet mold. We know, for example, that a sulphite and a groundwood pulp with equal and low Canadian freeness do not behave similarly on a paper machine The sulphite is slower in practice. The TAPPI drainage time of these two pulps reflects this difference correctly, and in my estimation, adequately for control purposes. TAPPI drainage is, however, relatively insensitive in the free range. This could be overcome when necessary, and I do not believe this would often be the case, by employing higher basis weight. The TAPPI drainage time has the additional advantage of (1) not being subject to high "wire" losses, such as often occur in the freeness tester, and (2) there is no "zero" limit to its description of slowness, and (3) the pressure drop across the pad is greater than in the case of the freeness tester. The instrument will therefore more readily pick up differences in pad compressibility.

A previous speaker mentioned his interest in handsheet porosity. The porosity of a dry sheet correlates very well with the TAPPI drainage time, indicating that the wet pad porosity plays a very large part in determining the dry pad porosity. Maybe we ought to simplify our speculations instead of making them more complex. We are always pulling water through a sheet no matter which drainage testing device we use, so how much differentiation can one hope to obtain by using similar experimental techniques? Maybe passing water through a sheet is very similar to passing air through a sheet. That brings up the next consideration: how much does a wet fiber assembly change upon drying? That is, what is the realignment of fibers? I have tried to observe this under the microscope by drying very thin handsheets on the microscope stage and I failed to detect large changes in structure, even with respect to fibril arrangement. I am a little disturbed about it because it doesn't fit preconceived notions, but I think there is a great simplification to be accomplished here, rather than a complication. The dry sheet reflects in many, many characteristics, the wet sheet behavior and structure.

Mr. Gallay: Thank you very much. I would like to have a look at the Brecht theory of limiting state or saturation for a moment, because we should have a look at the importance of what happens when you put in an admixture of chemical pulps with groundwood, and I know that some of you people were terribly surprised when Professor Brecht came out with the result that under certain conditions the addition of more sulfite, let us say, to the newsprint furnish, resulted in a lower wet web strength, and this is explained in this very pretty theory of saturation. In other words, getting the maximum wet density in the sheet is very much like getting some little stones in with some big ones in an aggregate. Any comments on this business of saturation in the wet state, so that you won't have a definite mixture, depending, of course, on the properties of the longer fibers and depending on the properties of the shorter fibers that you are going to put into this mixture?

Mr. Bersano: I want to point out a fact that the evaltion of groundwood alone is not sufficient. As you say, it is the mixing of sulfite and groundwood that we must evaluate. We want to make good paper and not good groundwood. Now, in my opinion about drainage, what happens on the wire is that owing to the very high suction you have from the table rolls in the first part of the paper machine, in a high-speed machine, there is a velocity of water going through the wire and owing to the lower mass of the fines, they tend to be transported more easily than the rest of the stock. Part of it goes into the white water and part form a vey thin film on the wire surface. Now, when this thin film is formed the quantity of water passes through that film is constant no matter which suction you have or what pressure you have. We have made tests that when a thin film of fines is formed you can increase the hydraulic pressure or you can have a higher suction but the velocity of passage of the water through the film is always the same. So, in the first part of the wire you have this thin film and on top of it you have a suspension of sulfite and groundwood, and that takes a very long time to drain, and when you apply more suction to the vacuum boxes or the suction couch, what happens? The only thing you do is that that film is marked by the wire and you have wire marks and at the same time the water on top of that film carryig the rest of the fibers tends to make streaks in the sheet formation. Now, in my opinion, what would be desirable to achieve would be to see some way of evaluating how much of those fines go immediately in contact with the wire. As a matter of fact we have made some tests on a normal newsprint sheet. We expected to have less fines on the wire side and that was not true. There are more fines on the wire side and less on the top side under certain conditions, and in that case we had trouble. The groundwood we must obtain in my opinion, for groundwood is something that, even if they are fines, and I believe Mr. Clark is right, provided they are long and fine, is alright, if they stick to the sulfite and the other fibers so that you have a thickening on the wire and not a division of fibers because, in that case you will have a combined sheet, one of much higher density on the wire side, and one of light density on the top side.

Mr. Gallay: Thank you very much, Mr. Bersano. Any further comments on this wet web strength? I have an interesting, very brief question from Mr. Yankowski of Kimberly-Clark Corp., where he asks, "With the advent of the pick-up felt is the wet strength test important?" Now, I don't know enough about the pick-up felt and its effect to know whether you are entirely out of the woods with all strength factors on the paper machine.

Mr. Yankowski: Pick-up felts allow you to run well with lower wet strength finishes. Is is known that southern pine groundwood has a much lower wet strength than northern spruce groundwood. Actually, when you make a kraft and southern pine groundwood mixture the wet strength of that is lower than northern groundwood and kraft or sulfite and yet the finished sheet of southern newsprint is a stronger sheet than the northern sheet, that is the Canadian sheet.

Mr. Clark: I think we must agree, without any question of contradiction, that the wet strength of the sheet of moist paper depends, primarily, on two things, first of all on slenderness, fineness, or wet flexibility; southern pine pulp is much coarser than northern pine and poplar. The second important thing is fiber length but after having enough length for a given diameter then you don't need much more. Surface tensions effects hold the wet fibers together. With a thick sheet where several fibers fibers together make up its thickness then the effect of slenderness is not quite so important so you might then have length as the main factor. May I just take a moment to admit to Mr. Brecht that in our discussion I may have given an unfair picture. "Slime stuff" very likely has more cellulose in its content than the "flour" but that still doesn't disturb the thesis that their cohesiveness is very likely the same whether the one contains a little more cellulose or more lignin. They are both new wood.

Mr. Gallay: So much for wet web strength. Let us assume that we have all these fibers set in the web and dried. How much resistance have we to stresses and through what mechanism does this resistance to failure through stresses arise? With chemical pulps, we speak of bonding and of frictional resistance to fiber separation, as well as of the strength of the fibers themselves. We have virtually all of the wood substance available in groundwood, but is the situation different here? Are the hemicelluloses, or more directly, free hydrophilic groups available on the fiber surfaces for bonding, or are they buried in lignin? We know that the entities of which groundwood is composed are far less flexible and supple than fibers of chemical pulp so that frictional resistance to failure will be less. Insofar as bonding is concerned, should we speak of a hot melt bonding of lignin or hydrogen bonding through carbohydrates. I wonder whether Mr. Clark would like to comment.

Mr. Clark: Mr. Gallay, that is a dangerous question because I am going to start right off by saying that as I get older I get more and more convinced, that the fibrage theory that James Strachan stressed first in 1926 is getting, to my mind, to seem more and more correct. If you will bear with me, and this is where he was misunderstood, fibrillation starts with visible fibrils, in the microscope, then microfibrilis that are visible only in the electron microscope down to micellar bundles and, further on, down to cellulose molecules. Now, in the case of chemical pulps, when you beat them you get a whole miniature tree from fibrils that you can see in the microscope away down and then, on each of the tiny twigs, you have the cellulose molecules sticking out. In the case of groundwood, because of the lignin present, you can't branch the elementary tracheid or fiber out into this miniature tree, and the surfaces produced are much smoother. In that respect they are more akin to glass fibers with which Mr. Gallay has worked and published a lot of interesting work. The same ultimate bonding mechanism is in play. In the case of a chemical pulp we have a well-branched Christmas tree affair with the possibility of interwinding the macro and micro fibrils and finally the cellulose and hemicellulose molecules. In the case of groundwood you don't have so many macro and especially micro fibrils and semidetached molecules. It is more than a question of more surface properties with adjacent molecules of semidissolved cellulose and hemicellulosic materials, hydroxyl groups form hydrogen bonds, and my thesis this afternoon pointed out that because in groundwood we can't get proper macro and micro fibrillation the question of fiber slenderness, or fineness, becomes more and more and more important, because the only way we can get bonding with these poorly fibrillated fibers one to the other is the flexibility that enables their being pressed together by surface tension and ultimately getting the hydrogen bonds to form as the water departs.

Mr. Gallay: I am not sure that I obtained a clear-cut answer to my question as to whether hydroxyl groups were actually available for this purpose in groundwood.

Mr. Clark: They are on any hydrophilic surface which has been in moist atmosphere.

Mr. Gallay: You do not think that they would be covered up by lignin and rendered unavailable?

Mr. Clark: No, they are too small to be covered up. I think they are on the cellulose surfaces sticking out of the lignin. I think that anything that you can wet has hydroxyl groups on the surface. Those are the ones that bind the surfaces of glass fibers together.

Mr. Gallay: Yes. I don't disagree but wanted to get your opinion. Mr. Campbell, would you like to comment on this matter?

Mr. Campbell: It seems to me that in the case of all papers we have to have bonding, and that bonding is hydrogen bonding. In the case of the groundwood, having a certain amount of lignin, most of the lignin is on the outside of the fiber. Once you break through and begin to fibrillate, especially if you do as Mr. Brecht says and work it so as to tease out the fibrils, I think you can get good, effective fibrillation with groundwood. And the cellulose of the groundwood is the part that does the bonding. Now that is pure guesswork, but I think it is reasonably well founded. But in the case of the groundwood you only have half as much cellulose available per unit of surface as you have in the chemical pulps. The great big trouble of all papermaking is that when we come to reform the bonds that we have broken in beating or grinding, it is a very haphazard performance and a great many of the bonds are completely useless. The only kind of bond that is really effective in producing strength is where a fiber, and by fiber I mean any piece of material that is long and slender, is bonded somewhere near each end to two other fibers. A bond that is formed singly, leaving the rest of the fibers sticking out, or a bond that amounts to a little, wee bit of cellulose sticking to an already existent fiber, is not effective at all. We have got to do a lot of studying of how we can improve bonding in formation, if it is possible. I don't know whether it is possible. But as it is now, we jam a whole lot of fibers together, we spread the fibers out in a suspension on the wire, we drain off as much water as we can, we press out as much as we can, and then we dry the rest, and bonding takes place, probably nearly 100% efficient in the sense that bonds are formed from all available surface, but very much less than 100% because most of those bonds are no use. They are not in the right place.

Mr. Gallay: Thank you, Boyd. Any other comments on this matter?

Mr. Clark: May I take a brief point, please. It is an interesting fact, that if you take an ordinary groundwood and wash it with a weak solution of caustic soda, the strength increases very measurably, 20 or 30% maybe. Why is that? Because, I believe, you dissolve away a little of the lignin and you enable little more molecular fibrillation to occur. Of course, maybe Mr. Gallay and others would say that that is nonsense, that what you really are doing is removing lignin so that you are getting more exposed cellulosic surface. I don't know. That is a matter which will have to be resolved. The

other interesting thing I mentioned today, is that if you add a little gum to a beater of sulphite pulp you increase the strength very measurably, again perhaps 20 or 30%. Put that same percentage of gum into the groundwood and you get practically no increase, indicating that the gum is merely aiding frictional forces, and only if a fibrage is present is the gum effective. If you have no microfibrils, as in the case of groundwood, or artificial fibers gum or starch is practically useless.

MR. GALLAY: Yes. Of course, the sodium hydroxide might also act to give you a small amount of swelling of the lignin itself, and that would get just a little gooey, and when two of those surfaces got together you would have some stickum there to stick them together, so that it wouldn't necessarily apply to cellulose.

FLOOR: Taking a standard spruce-fir groundwood and bleaching it with chlorine dioxide and not putting in any mechanical energy to it, you can increase its tensile strength fourfold, double its tearing strength and get its folding strength up to 3500 or so, all, of course, accompanied by a tremendous amount of densification and water logging. Now, I don't know if there is any significant fibrillation involved here, but I would attribute it almost solely to the removal of lignin and removing the restraining forces allowing the fibers to shrink and compact and get incongruity with each other.

MR. GALLAY: Yes. That also happens of course with neutral sulphite semichemical and the like.

MR. CLARK: Well if you look at those fibers you are talking about with an electron microscope, you will see they are covered with microfibrils. By silvering the fibers and looking at them with an ordinary microscope, you can also see the fibrils there. On an unbeaten fiber there are no microfibrils but remove the outer surface by any means and at once you get them. Take a groundwood and look at it under an electron microscope and there are very few fibrils. Treat it with chlorine dioxide, to remove some lignin and, immediately, the surfaces automatically are densely covered with microfibrils. It is the importance of those microfibrils that we are inclined to overlook.

MR. GALLAY: I wonder whether Mr. Clark has any definite data on the relative amount of fiber breakage in a groundwood sheet as compared with a chemical pulp sheet. Mr. Clark, you touched on that very briefly in your paper. Have you any data on that topic?

MR. CLARK: No, I am afraid not. I can merely quote Mr. Van den Akker's recent results in which he appears to correct my impression, at any rate, that there were only very few fibers broken in the case of a tensile test made on an unbeaten sulphite test sheet and he points out that there are a lot more broken than we think; the same thing may apply to groundwood but, again, as I showed in one of my tables, groundwood comes from woods that all have about the same tensile strength and the same eleasticity, so I don't think that the intrinsic strength of the groundwood makes a great deal of difference. Of course, those differences do have an effect on the grinding as to whether one wood grinds a more slender and longer fiber than another assuming both are of the same dryness. I think that the main influence of the strength of the wood is indirect.

MR. GALLAY: Thank you. Are there further comments in the field of dry strength of groundwood sheets?

PROF. BRECHT: We have recently carried out some research in this field which may be of interest at this juncture. This research related to the dusting properties of offset and other printing papers of great significance in the printing industry. An instrument was developed for the quantitative evaluation of this undesirable property and attempts were made at relationships with various factors in paper manufacture. Insofar as groundwood was concerned, we expected to get far more dusting with groundwood of higher freeness. Generally speaking, this was found to be true. The surprising fact arose however, that the increase of dusting with increased freeness was far higher for the fibers than for the fines.

MR. CLARK: But why not? You have a more open structure with coarse fibers, and with a more open structure and you can more easily shake the dust out. If you have a fine fibered structure it is compact and you can't shake the dust out.

PROF. BRECHT: In our tests where we had a very coarse flour material as fines, the dust increased, but the proportion of dust represented by this coarse flour material increased out of proportion, and this was unexpected. I think you have given the correct explanation, and it shows among other things, that conditions in groundwood are rather different than those in chemical pulps.

MR. GALLAY: My own experience in our laboratory has been that the dust is an agglomeration of all the components. It certainly is not confined to the fines, but contains also broken fibers, long fibers, etc. I do not know whether other workers in this field have had the same experience. Mr. Campbell, have you anything to add in rebuttal after all that has been said?

MR. CAMPBELL: I don't know about rebuttal but I would like to, perhaps, draw a red herring across this business of wet strengths and dry strengths and strengths generally. There is one property of paper which I think is of great importance and is very seldom measured, and that is the property of stretch. We consider wet strength in relation to the breakage at that point in the paper machine and we consider strength with relation to breakage down on the dry end, and we consider strength with relation to breakage on a printing press and I don't think strength, as such, has anything to do with it. In all cases, when paper breaks under those conditions it breaks because it has not enough stretch. Now, we know very little about stretch. What is known can be summed up, I think, in about two sentences. One is that stretch is not time dependent as is strength. If you put a load on paper, given time, almost any load will break a piece of paper, but it always breaks at the same stretch. The other is than any bit of stretch short of breakage that is imposed has a large part of itself permanent and there is less stretch left to break later on. Now perhaps, in spite of the fact that Steenberg and Ranel have done more work on stretch than almost anyone else and are, I think, responsible for setting up those two statements, the work of Steenberg and Ranel and the others under the head of rheology of paper, is I think, responsible in part for our not knowing as much as we should about stretch because much of that work has been interpreted in terms of rheology and has been demonstrated by such analogies as dash pots and springs and even a theoretical viscosity of paper, although there are some doubts about that among some of the rheologists. Now, rheology is a science which deals with materials which flow, and it seems wrong to include paper in that class. Paper is a structure and the fundamental study of its properties must allow for this. Consideration should be concentrated on the elements of the structue, their arrangement, and their bonding. No one would think of doing otherwise in the cases of such structures as bridges or ships. And the reason structure plays such a small part in the interpretation of paper characteristics is that no one knows very much about its structure. It seems to me that someone should start to study the structure of paper. The bonding is a very important part of that. It can be reasoned that the bonding between two fibers pointing in the machine direction is much stronger than the bonding between two fibers at right angles because, during the drying, the tension doesn't have anything to do with the bonding between those fibers in machine direction, but it does put a limit on the bonding in the other way. I don't know if that is an area where we know very little and it is time that somebody did some studying. I would suggest that, as a first, very primitive experiment, someone should take a very thin sheet like, say, a paper tissue, and stretch it under the microscope and watch what happens to the bonds. You can

see them under a microscope, or if you project them you can even take a photograph of them. Some of that work is probably going to be done as an outcome of the work that Van den Akker is doing and has published in a recent issue of *Tappi*, where he was primarily interested in seeing how many fibers pulled out whole and how many were broken when a sheet was broken. I think if he keeps on the way he is going, he is going to begin studying paper structure.

Mr. Gallay: I am glad you brought up this matter of stress-strain. I would like now to turn to the fount of most of our wisdom in this field and ask Professor Steenberg to continue with this phase of the discussion.

Mr. Steenberg: Thank you. As a matter of fact I have written a couple of papers on this subject, but I believe I have never used the word "rheology." On the other hand, rheology has in the last years become a term used in connection with all sorts of deformations of matter which do not proceed entirely Hookean, for instance even production of slip lines in crystals or twinning of crystals under pressure. As far as I remember the term rheology of paper was introduced by Stan Mason in 1948. Personally, I think the term is dangerous to use among papermakers, because they will immediately think about viscous flow, which in their minds has a definite physical interpretation.

The aim of the stress-strain studies on papers we have made is to increase our knowledge about how paper behaves, of course also groundwood paper. In order to condense the acquired knowledge a comprehensive theory is needed, from which the different relations can be obtained without memorizing them all. The theory is mathematical but in order to make the theory still more easily remembered, it is depicted by a model. In stress-strain such models are usually mechanical models made up of dashpots and springs. These models do not explain how paper is built up, but they depict in an easily remembered way its behavior. Because the dashpot model contains viscous elements the viscosity concept has entered into too many minds. If instead the mathematical theory was illustrated with electrical models, the concept viscosity would not have entered the picture. The analogous electrical models could have been chosen in the first instance just as well, but they are not so simple to understod as mechanical models. Because the only aim of the model is to make things more easy to depict they are consequently less useful.

Personally, I still think that there is no method superior to the mechanical model of paper if I have the aim in the shortest possible time to give somebody a condensed qualitative knowledge about the behavior of paper under stress and strain. With some knowledge of the model he can later find out for himself a great number of things which would have taken a series of lectures to tell him, and which he probably would have forgotten when he needed the information.

All theories and model conceptions are white lies; they could be used as long as they are useful, and dropped the very moment they get useless. For many purposes it is, for instance, useful to assume light to be made up of bullets of photons, in other cases it is more convenient to assume light as a wave motion. Both are pictures and let us use them when useful but not be deceived by the physical analogy used.

What is needed today is not discussions about vocabulary or shortcomings in different model conceptions but a more detailed knowledge of the actual elementary processes of deformation of a web of paper. This cannot be solved except if we invent a method which will trace what happens inside the paper and which can definitely show the types of processes that occur.

Important contributions to such methods have been given by Van den Akker and Nordman. In our laboratory J. Kubát has studied electrical noise in paper ring staining. In principle any electrical conductor has small variations in its electrical resistance. If these fluctuations are amplified they can be heard in a loud-speaker if the frequencies are in the audible region. It has been found that the electrical resistance of a paper fluctuates very markedly during stressing, producing a high electrical noise level. This noise does not, however, proceed during relaxation processes, and, as a matter of fact, a complete loop in a stress-strain cycle can be passed without noise production. As soon as the stress is higher than that of the top of the loop, noise production immediately starts. This shows that there are at least two different mechanisms in the deformation of paper.

I hope that many more paper researchers will try to find methods which will increase our knowledge of the basic processes in paper deformation.

Mr. Gallay: That was extremely interesting, and I am glad that both of you gentlemen have placed rheology in its proper sphere because the word, of course, is dangerous in the concept that we use it, and we should stick to "stress elongation" which is rather more understandable in the context. Anyone else like to make any comments or ask any questions on this matter of dry strength?

Mr. Tuck (Howard Smith): In talking about paper strength there was one thing I was hoping either Mr. Steenberg or Mr. Campbell would mention then they started talking about various factors that are important. There is one problem I got involved in over a period of time, and the only factor that was important there was the work of rupture, which is the area under the stress-strain curve.

Mr. Steenberg: I don't think one can say more than that, for paper under impact, the work of rupture (the area under the stress-strain curve) is the property of greatest practical interest, at least among those which are easily determined. As far as newsprint is concerned I am not quite sure this work of rupture, determined from a quasistatic pendulum tester, is relevant. I believe that the rates of impact on newsprint in service are so high that any static measurement is of relatively low significance. At any rate I don't think breaking strength is of importance for newsprint. As a matter of fact, newsprint in Scandinavia has little more than half the tensile strength it had 25 years ago, and I don't think any modern pressman would like to run the old type of paper with high tensile. Pliability of newsprint is a very important property and this is in all probability related to relaxation processes, the rate by which stresses can be relieved and distributed. This property is of course also related to the stress-strain curve but not necessarily the work of rupture

Mr. Clark: I hate to agree publicly as I do in private with my old friend Mr. Campbell, but stretch is, no doubt, the key test as far as paper is concerned as to its running qualities as a part of the thesis of my paper today, how do we make a groundwood sheet stretch more? One of these answers, we make the fibers finer, more slender. If we do that we can't help but increase the actual area of paper which is involved in a sheet occupied in absorbing a given stretch. If you can imagine that the fibers are very, very coarse, perhaps like tiny wooden matches, the sheet would obviously have no stretch at all. If you can imagine the cellulose fibers to be very, very fine in the nature of submicron glass fibers for example, then you get a lot of integrity in your sheet structure and a lot of built-in stretch. Since the flexibility of the fibers is inversely dependent on the fourth power of their diameter, if you reduce their slenderness the applied load is better distributed and more work has their slenderness to be done to break the sheet. I have no doubt that Mr. Steenberg would agree with this thesis too, and perhaps also with the fact that the appreciable noise that you get when you stretch paper is caused simply by the rearrangement of the individual fibers under the imposed load. Once the crust, so to speak, is broken the noise quietens; it is like putting a stress on a starched piece of cloth. After the fibers take up the stress then you have very little quietness.

Mr. Gallay: Any comments? If not, we might touch for a few moments on another factor which I mentioned at the outset, namely, groundwood in relation to printing properties. We all recognize that the compressibility conferred by ground-

wood content is highly desirable in letterpress printing. I wonder whether anyone would like to comment on smoothness, ink receptivity or any other printing factor.

MR. CLARK: Again, I want to point out that, as far as printing and printability are concerned, particularly with a uniform kind of pulp such as groundwood is with respect to its surface properties, that the printing quality resides very largely in the slenderness of the fibers. It is quite obvious that if you have coarse fibers you are not going to have a good printing surface, and that is one reason why southern pine, for example, doesn't print as well as northern pine, other things being equal. If you can increase the fineness of your fibers then you get a better printing surface. I just want to reiterate that fiber slenderness, now that we can measure it, should become very important, transcending the whole gamut of, use properties of groundwood.

MR. CAMPBELL: I think you stuck your neck out there, Jim. Newsprint is pretty good for printing on but glassine isn't.

MR. CLARK: Glassine surface doesn't have the pores that the groundwood has, which causes its increased activity with respect to ink reception. I prefaced my remarks by referring to the same kind of pulp. Now, with glassine pulp on one hand and groundwood on the other, we have two different animals.

MR. CAMPBELL: Well, if you get your groundwood very, very fine, then it becomes like a glassine.

MR. CLARK: No, that doesn't happen, the structure will not become as compact.

MR. GALLAY: I see that the hour is approaching 6 o'clock. I would suggest that we have a few minutes for a question or two from either the panel or the audience. Mr. Clark has a couple of unanswered questions.

MR. CLARK: Here are the questions. I haven't read them yet. How would you expect to be able to correlate a product with freeness when you have so many interfering variables such as headbox temperature, speed, percentage sulphite, quality, formation printability, wire mesh, wire length, at headbox, open or pressure type of pick-up, vacuum, or open quality of sulphite, and so forth. Well, I guess the answer to that is that you just wouldn't. But this is in reply to this indirectly, I defy any one to show a real, valid correlation of data between machine speed and freeness, and I don't care what machine you have. At least, if you have it, for goodness sake bring it out, because I have been hollering for it for years and years. The freeness test, gentlemen, is mainly a measure of the amount of debris in a pulp and nothing more. It is a very useful guide for wood grinding, and I would certainly recommend it for that purpose but, like my friend George Underhay said at the Cambridge meeting last year, why drag a mongrel pup into Cruft's Dog Show? We shouldn't talk about freeness when we are being scientific. Freeness is the sort of test that should be confined to the mill, mainly as a conversation piece, and it is fine for that purpose, but when we start relating things to freeness, matters get awfully complicated.

MR. GALLAY: Thank you very much. In concluding this session, may I say that my colleagues on the panel and I have enjoyed this session very much and we hope you have also. We thank you for having been a very good audience. The session now stands adjourned.

Fourth

Mechanical Pulping

Conference

Edgewater Beach Hotel

Chicago, Ill.

Sept. 19–21, 1961

Advantages of Individual Grinder Pit Testing

FRED W. BISHOP

SOUTHLAND Paper Mills started up experimentally with practically no background of experience in the production of newsprint quality groundwood from southern pine. With a limited number of stones it was necessary that drastic changes be made on individual stones to study the effect on pulp quality and to control the quality on the blend of pulps from all stones. It was also necessary to evaluate stones of various grit, hardness, composition, and the effect of various burrs for optimum quality. It was essential to evolve a sampling and testing procedure in which the operators could have some reliance regarding the quality of stock produced from each grinder pit.

It was established by this early work that for the purpose of this mill a composite sample representing seven independent samplings, 30 min. apart, was generally representative of the pulp being produced by that stone under a given set of operating conditions. Consistency and freeness have always been run on each stone composite and in the early days, handsheets were made and mullen and tear were run on these. In the past as many as two composites were collected per shift in order to have two freeness tests on stone operation during that period. More recently, with in excess of twenty stones in operation, only one composite per stone per shift is collected for test purposes. Samples from each of the three shift composites are collected into a 24-hr. composite which is used for handsheet making for mullen and tear tests.

Table I. Pit Consistency, %—One Day of Data from Fig. 1

Stone	Time 8–12	12–4	4–8	8–12	12–4	4–8	Av.
1A	..	426	..	478	..	500	..
1B	..	510	..	431	..	510	..
2A	..	307	..	371	..	442	..
2B	Under Repair						
3A	..	484	..	498	..	470	..
3B	..	478	..	497	..	487	..
4A	..	450	..	425	..	460	..
4B	..	440	..	432	..	421	..

All of these stone data are posted as produced for the benefit of the grinder operators and their supervisors. The posting chart is set up to include the data for all stones in one mill for a period of one week. The collected data for each day are incorporated in a Groundwood Mill Production Report for the benefit of the superintendent where additional information covering the operation of each stone includes such figures as tons per stone day, kw.-hr. per ton and mean kw. per line. The groundwood mill supervision takes into account all of these factors on any one stone before they take any steps toward changing a stone face to bring it back into proper operation. Rarely ever are they guided by the results of one

FRED W. BISHOP, Technical Director, Southland Paper Mills, Inc., Lufkin, Tex.

Table II. Freeness, ml., C.S.—One Day of Data

Stone	Time 8–12	12–4	4–8	8–12	12–4	4–8	Av.
1A	..	77	..	76	..	80	..
1B	..	64	..	45	..	59	..
2A	..	32	..	33	..	38	..
2B	Under Repair						
3A	..	64	..	42	..	40	
3B	..	45	..	55	..	37	..
4A	..	65	..	45	..	52	..
4B	..	63	..	51	..	59	..

WEEK

GRINDER PIT TESTS

TESTS BY TECHNICAL SERVICE DEPARTMENT

	SUNDAY							MONDAY							TUESDAY							WEDNESDAY							THURSDAY							FRIDAY							SATURDAY						
	8-12	12-4	4-8	8-12	12-4	4-8	AVG	8-12	12-4	4-8	8-12	12-4	4-8	AVG	8-12	12-4	4-8	8-12	12-4	4-8	AVG	8-12	12-4	4-8	8-12	12-4	4-8	AVG	8-12	12-4	4-8	8-12	12-4	4-8	AVG	8-12	12-4	4-8	8-12	12-4	4-8	AVG	8-12	12-4	4-8	8-12	12-4	4-8	AVG
	CONSISTENCY																																																
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Fig. 1

Table III. Mullen lb./sq. in. on 32-lb. Handsheet—One Day of Data

Stone	Time 8–12	12–4	4–8	8–12	12–4	4–8	Av.
1A	..	..	..	..	..	..	4.0
1B	..	..	..	..	..	..	4.0
2A	..	..	..	..	..	..	5.5
2B	..	..	..	..	..	..	..
3A	..	..	..	..	..	..	4.6
3B	..	..	..	..	..	..	5.9
4A	..	..	..	..	..	..	5.7
4B	..	..	..	..	..	..	5.9

day's reporting, but in general their actions are governed by a trend of results from a number of days.

Figure 1 is presented primarily for the purpose of illustrating the type of posting chart which consolidates the results of one week's operation as the tests are produced for the benefit of the operators. To point up the specific items as tabulated in Fig. 1, the individual data for a day are extracted and tabulated in Tables I, II, III, and IV. The extent of test variations over a period of one day in groundwood mill no. 1 are shown.

The average data from the first four tables are assembled with additional data giving a picture of a single groundwood mill's operation for the benefit of the groundwood mill superintendent and a tabulation of these data is shown in Table V. For simplification, the average data from the first four tables have not been listed in Table V. It should be pointed out that all groundwood production is prorated to each producing mill and to each individual stone in that mill

Table IV. Tear, grams for 16 Ply of 32-lb. Handsheet—One Day of Data

Stone	Time 8–12	12–4	4–8	8–12	12–4	4–8	Av.
1A	..	..	..	..	..	..	15.6
1B	..	..	..	..	..	..	17.0
2A	..	..	..	..	..	..	16.0
2B	..	..	..	..	..	..	...
3A	..	..	..	..	..	..	14.4
3B	..	..	..	..	..	..	16.0
4A	..	..	..	..	..	..	15.4
4B	..	..	..	..	..	..	16.9

by actual stick counts. In the first column opposite stone number, the stick count per stone is listed and the production for that mill is prorated to the stone by the number of sticks used. This stick count to the individual stones is accumulated by the grinder man as he charges each pocket of a stone by counting the number of sticks in each charge and immediately tapping off the count on a totalizing counter. These counters are read at the end of each shift and the 24 hr. total represents the stick count for each stone. In the case of this mill, the stick count method for allocating groundwood production has been an accurate method for this purpose. On first thought, it would appear that all sticks must be of practically the same diameter but this is not the case as the wood being used over a 24-hr. period generally will average out in diameter as it is fed to each stone.

Table V. No. 1 Groundwood (Gwd.) Mill Daily Report

Stones	No. of sticks	Tons no. 1 Gwd.	Tons/ stone day	Mean, kw.	Kw.-hr.	Consistency, freeness, Mullen, Tear
1A	1,299	15.4	19.5	2650	1600	..
1B	1,594	18.9	18.9			
2A	1,510	17.9	17.9	2706	1750	..
2B	Under Repair					
3A	1,974	23.4	23.9	2790	1280	..
3B	2,041	24.2	24.2			
4A	1,738	20.6	20.6	2590	1520	..
4B	1,696	20.1	20.5			
..	11,852	140.5	20.8	2700	1520	..

Table VI. Data Presented in Monthly Report

	Position →	1A	1B
	Number →	5521	7573
	Class →	37C601-07V	37C601-07V
No. of times	Sharpened	2	7
	Bricked	0	1
	Turned	0	1
Stock tests	Consistency	4.49	3.87
	Freeness	57	39
	Mullen	5.3	5.9
	Tear	17.4	17.8
Stone days		14.31	14.92
Production (tons/stone day)		20.73	20.35
Stone cycle factor		392	304
Power (kw-hr./ton)		.. 1570	..
Load/line (kw.)		.. 2690	..

All of the daily data shown in Table V are finally compiled into a monthly average figure which is shown in Table VI. For the purposes of this paper the data are being presented on one line of grinders and cover two-stone operation. There have been occasions in the past operation when continued monthly examination of these data indicated the necessity of removing as many as two stones from a line due to high kw.-hr. per ton, low production rate and poor quality. The monthly averages truly point out any faults of stone operation that continue to be significant.

This system of individual stone analysis has been of particular advantage in the evaluation of the Norton Co. G-bond stones. Two of these stones were installed on one line a number of years ago and it soon became apparent that the pulp from these was being produced at a higher production

Table VII. No. 8 Line as Compared to No. 1 Groundwood Mill

Month	Kw.-hr./ton	Tons/stone day	Freeness
Nov. '55	−240	+3.88	−14
Dec. '55	+ 70	+1.36	−26
Jan. '56	+ 10	+0.66	−23
Feb. '56	− 90	+1.63	−18
Mar. '56	−160	+2.59	−15
April '56	−190	+3.47	−11
May '56	−230	+3.75	−12
June '56	−140	+1.97	−19
July '56	−170	+1.81	−17
Aug. '56	− 70	+0.41	−26
Sept. '56	−110	+1.37	−30
Oct. '56	− 90	+1.34	−22
Nov. '56	−190	+3.18	−23
Dec. '56	−110	+1.73	−32
Jan. '57	−300	+4.81	−14
Feb. '57	−190	+3.27	−19
Average	−138	+2.33	−20

rate, lower kw.-hr. per ton and surprisingly enough at a lower freeness than pulp produced by conventional stones. Tabulation of typical data from these two stones is shown in Table VII. This table lists the comparative differences between the no. 8 line of no. 2 mill and the averages from four lines of no. 1 mill where all vitreous bond stones were in use. As a result of this good experience with G-bond stones, a total of ten such stones are operating in a possible 26 positions. Without exception all of these stones have continued to perform in the same manner as the first two.

All of this sampling, testing, and data handling appears to be laborious and time consuming, but once the routine is established the compilations are quite simple. Two men per shift, namely a sample runner and groundwood tester, produce and post all of the physical stock tests. A clerical worker devotes approximately 20 hr. a week to the assimulation of data and the final reporting.

The proper association of these data with blue glass examination of pulp from the individual stones is a very good method for evaluating stone face condition. All of these data reflect the operator's efforts to maintain stone operating standards and pulp quality.

Panel Discussion: Optimum Grinding Conditions

CHAIRMAN WHITMORE: To start off I would like to introduce our first speaker, J. J. Kirkpatrick, Bowater Southern Corp., Tennessee, who is going to discuss his micrometer burring.

MR. KIRKPATRICK (*Is Micrometric Burring Worth While?*): We have heard many operators say that you cannot sharpen stones micromatically. Now, I will try to point out a few reasons why you can. We have sharpened stones by the sense of feel and I know that two men will not have the same sense of feel. One man may hit a stone too hard and the other not hard enough. In my own personal opinion, you are only guessing when you sharpen by a sense of feel only. Possibly, you may have a man in your mill who is on the payroll, and does a pretty good job of burring using a good sense of feel. If so, he is a rare bird. Those fellows are few and far between. We have been dressing stones micromatically for several years and know that the end results are much better and more foolproof than sharpening by guesswork. When you sharpen a stone micromatically, you make adjustments for that purpose, for the desired penetration, the burr's positioned half on and half off the stone with the load taken off the grinder. After the burr is positioned it is slowly lowered until a spark is seen denoting contact with the stone. After this spark is seen, a notation is made of the micrometer reading and the burr is raised slightly off the stone so as to pass over it and not wipe off the stone pattern. While the burr is taken off the stone face, using a noted micrometer reading as a zero point, adjustment is made to the fork depth for a desired penetration and the lathe is then started across the stone, which gives you the same pattern on all rows of stone segments. We have visited mills all over North America, and we have never been able to see a full pattern on all the rows of the stone segments unless the stone was sharpened with the micrometer lathe. When you sharpen by sense of feeling, the burr tends to stay in one position too long before you run it across the stone, thus wiping the pattern off right at the starting point. Soon the stone will become rather round because of the increasing amount of stone being rubbed off at the point of shortening each time. Stones should never be sharpened by unsupervised personnel. Stones sharpened by unsupervised personnel will result in loss of stone and excess cost and reduction of stock quality. Now, I shall try to point a method that we use down in Calhoun, in regard to micromatically burring of stones. First thing we do is to check our lathes and we do this by checking the alignment of the lathe in three different positions. The lathe has been aligned with the center line of the shaft. When it is perfectly in line and level we dowel the frame of the lathe in that position, that is, the relation between the lathe and the stone always being parallel. You cannot expect to get a product any better than your lathe will allow. Lathes should always be checked for level for each stone turning and we do that in all cases. Try to have each burr holder, bushing, and pin as near perfect as you can, and both the i.d. and o.d. measurements for each individual grinder. Never use a complete burring unit except on one grinder. We drill holes in the bushings to help the lubrication while the burr is in sharpening use, and we use a graphite grease or a grease of comparable nature. We check our newly purchased burrs often to see if they are out of round and, if so, we notify the vendor and allow him to check up his inspection personnel. Never use a burr too dull, as it will have a tendency to break your pattern and the stock value will be lowered. When you true up a stone, whether it is new or old, you get a burr reading when the stone is cold or in a comparatively cold condition. You should not try to use this reading as the initial reading for all sharpening purposes. Get a new reading each time when the stone is up to the desired temperature, to use as a zero reading for your future sharpenings until you change the burr. Always get a new zero reading when the burr is installed on the lathe. Now, we find differences in initial reading will occur in the following magnitudes for the following reasons: a new burr versus an old burr will vary between 20 and 40 thousandths, a hot stone versus a cold stone will vary in the range between 30 and 40 thousandths. We sharpen mostly with 4 thousandths penetration, occasionally 5, depending how deep our pattern on the stone is wanted. We find it much better to sharpen light and a little more often rather than deep right at the start. If we don't get the desired results with the light pattern we go back and apply another 4 thousandths cut. We inspect stones weekly, look for burns, pattern, joint material, and mechanical breaks and beveled edges. We arrive at what stone needs turning, first by visual inspections and also bevel at the same time if it is needed. We believe, to get good quality and runnability for our paper machines, we must be very particular with the dressing of our stones properly, and with the equipment in as good condition as humanly possible and keeping this equipment properly maintained.

CHAIRMAN: Our next speaker listed on the program is Bill Dunphey from Anglo-Newfoundland. However, he was unable to make this trip and Steve Steeves is taking his place and discussing the effect of surface speed on pulp quality.

MR. STEEVES (*Effect of Surface Speed on Pulp Quality*): With the paper machine speed-up programs which have been relatively general throughout the industry, many groundwood mills are faced with the problem of increasing their output. When additional grinders are necessary, one of the most contentious decisions which must be made is, what should their surface speed be? There appears to be a general opinion among groundwood personnel that better control and quality are possible with lower speeds, in the order of 3500 to 4000 f.p.m., yet published experimental work, while often in conflict, seems to indicate that satisfactory quality groundwood can be produced at higher speeds and, correspondingly, increased production rates per stone.

In the 1930's four important investigations were published. Johnson and co-workers reported only slight reduction in specific energy consumption and quality as surface speed was increased, both with a semicommercial and a miniature grinder. Brecht, on the contrary, found quite considerable reduction in specific energy consumption and quality, while Schaffer and Pew reported specific energy consumption to be independent of speed and quality to be somewhat lower at the higher speeds. With a commercial pocket grinder, McAdam produced satisfactory groundwoods at speeds of 5400 and 6000 f.p.m., but the average fiber length was reduced. In 1942, Jones and Holland, in a continuation of the work of McAdam, operated an artificial board grade stone at 5400 f.p.m. Similar groundwood at the same specific energy consumption was produced as with normal sandstones at 3200 f.p.m. This was with a pocket grinder. The Roberts grinder, however, running at 5400 f.p.m. only operated similarly when a stone but slightly coarser than normal was employed.

Ten years later Collicutt and Cochran operated a commercial Roberts grinder at 4400 f.p.m. and a miniature grinder up to 5000 f.p.m. The quality was slightly lower at the higher speeds when compared at the same freeness. With higher speeds a duller stone or lower grinding pressure was necessary to obtain the same freeness.

The question of surface speed was again revised in recent months with publications from both Steenberg and Brecht, in which the general tendencies are in close agreement. In the range of 1600 to 3500 f.p.m. Brecht found that production increased considerably with but slight reduction in specific energy consumption. In the range investigated any pulp property could be maintained constant by employing the correct stone sharpness. At constant freeness,

however, strength properties were somewhat lower at the higher speeds.

Although the investigations which have been published are not in complete agreement, some general conclusions may be drawn. As surface speed is increased, production increases in approximately direct proportion, specific energy consumption remains relatively unchanged, and quality is somewhat lower when compared at the same freeness.

Experience at our mill has not been extensive with various surface speeds. With three pocket grinders having 54-in. diam. stones, the most suitable speed was 3100 f.p.m. When the speed was increased to 4200 f.p.m. the stones became much more difficult to control and the burst dropped 20% for the same freeness. Yet today we are operating Great Northern supergrinders having 67-in. diam. stones at 4200 f.p.m., and producing satisfactory groundwood without experiencing undue control difficulties.

It is thus, unreliable to use data established for one grinder to predict the results to be obtained for another. The interrelationship between surface speed, other grinder variables, and grinder design is not yet clearly understood. One factor, for example, which has been neglected is the relative amount of refining which takes place between wood and stone. This is certainly dependent on the type of grinder, length of grinding zone, arrangement of showers, etc. More information in this respect is required and could, possibly, account for some unexplainable observations which presently exist.

Let us briefly consider the initial defibration of the wood as influenced by surface speed. According to the theory of Klem, an optimum speed exists for any particular length of grinding zone, which will allow a grit to just pass the grinding zone in 50 to 60 milliseconds. In this time the wood should advance a length equivalent to one fiber diameter. Under these conditions a maximum amount of good fibers are initially produced, since plasticization of the lignin bonding occurs. It has been pointed out, however, that it is possible to produce any grade and quality groundwood with laboratory and miniature grinders which have lower grinder zone temperatures and shorter initial grinding time. This would indicate that plasticization of the lignin may be of minor importance.

Perhaps the theory of Cambell more closely represents what actually takes place during defibration. This states that a crushing or fatigue mechanism is possible. Under normal grinding conditions the wood is struck blows with a frequency in the order of 6000 per sec. by some 150 lands which must pass the wood for the removal of a depth equivalent to one fiber diameter. With higher speeds, as suggested by Brecht and Weiss the frequency is correspondingly greater. The mechanical action between wood and stone is more severe and the elastic limit of the fibers more frequently exceeded. This results in more broken and shattered fibers and the quality of the initially produced groundwood drops. Providing other conditions are maintained constant, however, there is a partial quality compensation as more ground wood is carried back from the pit to the grinding zone for further refining.

In summary, it is felt that an optimum range of surface speed exists for any particular grinder and set of grinding conditions. The magnitude and initial expense necessary for any single investigation setup to gather these data makes such a project impracticable. There are, however, throughout the industry, similar grinders operating at slightly different speeds. Valuable information might be available if data could be compiled under otherwise similar conditions and forwarded to a central group for analysis. With these data it might be possible to answer such questions as: "what is the optimum speed for our existing grinders?" "Would it be desirable to convert to another speed for economic or quality reasons?" "What speed should be installed with new grinders?"

CHAIRMAN: The next talk is by J. H. Perry, Norton Co., on a very pertinent topic.

MR. PERRY (*How Much Power Should Be Connected to the Stone?*): How much power should be connected to the stone? I do not know. No one else knows. However, let's review just a bit just what has happened in industry since the beginning of grinding. We heard from one of our good speakers yesterday that in the early days of grinding, 10 hp. was considered a sizable figure. Great progress was achieved when they got up to 100 hp. per stone. We have mills now, in the groundwood industry, hp. with power inputs as high as 7200 hp. per stone. In making this statement, it should be pointed out that this is a very large stone and rather than consider simple power inputs we should consider the actual square inches of grinding area on the stone face. Where 7200 hp. is being used, this amounts to $1^1/_2$ hp. per sq. in. of grinding area. There are mills in Canada, I know, two at least are operating at 1.4 hp. per sq. in. The answer to the question, how much power should be connected to the stone lies, in my opinion, in this area. We do know that under these conditions of grinding in the 7200 hp. area there are times when the power applied per square inch must be far in excess of the $1^1/_2$ hp. We know, for example, that when one pocket is empty the pressure is increased in the remaining pocket to absorb the power input of the motor. It is estimated that the power in this case amounts to 2.7 hp. per sq. in. While this is not a standard or normal condition, it does exist at times, at least every time a pocket is loaded.

So, on that basis, I would say that we are safe if we design to absorb 2.0 hp. per sq. in. We have no fear of the stone performing satisfactorily under such conditions, provided, of course, that the grinder has the showers properly located and there is a quantity of water sufficient to cool it and to transport the pulp away. The grinder must be so designed that the quantity of pulp produced at this high power input will readily flow away from the stone. There are mechanical limitations with respect to power input for all pulpstones. As you know, we hold and drive the stone with pure friction in the flange bearing area. To increase the power input we must increase the flange pressure and then we expose ourselves to the danger of the concrete in the flange bearing area crushing and thereby causing the stone to slip, or it may not be possible to even drive the stone. To put more power in the present grinders, the shaft diameter would have to be increased. This then, imposes another problem on the stone manufacturer. Increased shaft diameter means that we have to make the hole in the stone larger and in some cases would have to sacrifice some of the steel reinforcing that is essential to it. The present grinders and equipment are working well. With the present design of grinders and pulpstones the limitation to higher power lies in surface speed. The pulpstone manufacturers, over the years, have recommended speeds not higher than 5000 surface f.p.m. At this speed we know that we have a safety factor of 5.8. As the speed increases the factor drops until, at 10,000 surface ft. there is a factor of only one. From an engineering standpoint, a factor of one is not adequate. Conceive for a moment, what would happen if the stone burst at high speed. The grinder would be wrecked, and undoubtedly people would be injured. With stones constructed in the present manner, the safety factor of 5.8 at 5000 surface f.p.m. insures the mills of adequate safety and dependability. If you are contemplating putting more power in your grinders, I believe you should consider it from an efficiency standpoint.

Such a move would result in more economical operation and more uniform pulp. Control of pulp quality becomes more critical as power is increased and the skill of the operator is tested to maintain uniformity. Under these optimum conditions changes can occur quickly and they frequently do. Other than the limitation of surface speed from the safety standpoint I think we have not reached the limit of power input at this time.

CHAIRMAN: Our next speaker, A. M. Cuthbertson is from Abitibi Power & Paper Co. Ltd.

MR. CUTHBERTSON (*What Is the Ideal Grinding Pressure?*): I have been asked to sit on a panel to discuss optimum grinding techniques before I knew what my 3 to 5-min. topic would be. When I learned that it was to be "What Is the Ideal Grinding Pressure" my first and last reaction was to ask myself—What is the ideal grinding pressure? Is there an ideal grinding pressure? How do we know an ideal grinding pressure?

Let us look at the following formula: hp. $-0.000007933dN(\mu W)$ which is a modification of the prony-brake formula, where d = the diameter of the stone in inches; N = speed; $-W$ = total thrust of foot against the stone's surface less friction loss and lateral pressure, and 9 = coefficient of friction between the wood and the stone's surface. For our purpose it could be written thusly: Hp. = $K\mu W$.

Now if there is an ideal grinding pressure under our control, it would determine the exact size of motor required to drive a line of grinders; production would be constant; horsepower would be constant; freeness and strength would be constant; the per cent tailings would be constant; refining maintenance would be cut to a minimum and consistency of deckering would be constant, thus cutting the required size of decker chests and simplify consistency regulators. The use of a master governor to control total mill load would be at the sacrifice of all this. This would be especially so in a mill where only half their grinders were driven electrically and large daytime converting operations make the task of the master governor quite difficult. However, in most mills the master governor is a necessary economical installation. There goes one constant!

Let us look at μ, the coefficient of friction between the wood and the stone's surface. I have had little experience with trying to measure the coefficient of friction but needless to say, with various types of wood used in mills and wood from widely scattered localities and with the constant, if slowly, changing surface of the stone, the coefficient of friction is bound to vary, moreso in some mills than others. Even the coefficient of friction from one grinder pocket to another is different.

W = the total thrust against the stone's surface less friction and lateral pressure. In three-pocket grinders where filling a pocket means arranging the wood to keep binding at a minimum, it seems that where speed charts, load charts, or governors are not installed, the lateral pressure approximates the total thrust of the piston until pockets are shaken up. In considering the bigger automatic type of grinder it has seemed to me, and no doubt there will be many who disagree, that there is greater binding in the Great Northern type of grinder than in the magazine type. Nevertheless, there is more binding when the pressure foot is on the last 2 to 3 in. of its stroke, than the beginning of the high pressure cycle. This point is necessary to mention as operators are not given instruments to measure the actual pressure of the wood on the stone's surface but only the cylinder pressure in pounds per square inch.

However, let us presume that we have a constant total thrust of wood against the stone. How much bearing would this constant pressure have on stock uniformity? We are dealing with cylindrical belts where the total area of wood against the stone is constantly changing, resulting in a constantly changing unit pressure. We are dealing with sapwood, heartwood, spring growth, and summer growth, which add to the variables encountered.

In summing up, I wish to point out that in the groundwood mill we are dealing with a product of nature. There is one thing we know—nature does not make a uniform product, but we are given the task to make a uniform end product. Why not use all the tools at our disposal? One of these tools is the grinding pressure.

Let us ask ourselves one more question—"What is the ideal grinding pressure range and what are its limiting factors?"

R. MURLEY (*Optimum Grinder Pit Conditions*): Optimum grinder pit conditions are those which produce more favorable conditions in the grinding zone and hence result in the production of an improved groundwood. Generally speaking, they are conditions of high pit temperature, low pit consistency, low stone immersion, proper pit design, and arrangement of showers. Other factors being equal, adherence to the above conditions can be said to show a trend toward improved groundwood, characterized by a high percentage of long flexible fibers, high burst, high tear, and good drainage. There is also evidence of slightly increased production, and slightly decreased energy consumption.

In my opinion, optimum grinder pit temperature is 185 to 190° F.; optimum pit consistency, 1 to 2%; optimum stone immersion, 2 to 3 in.

I think the thought which is in most of your minds right now is "What's so different about these conditions? Why, in our mill we're operating very close to those conditions right now and we're still not satisfied that we have the best groundwood that we are capable of producing." If such is the case, it is well to remember that adjustments made in the grinder pit are only refinements which, if in the right direction, show a trend to improved groundwood. After all, the grinder pit is not the grinding zone and changes made there, unless extreme, are only minor compared with such other often uncontrollable factors as, depth of sharpening pattern, wood condition and species, the varying angle between stone surface and wood, etc. For most mills, adherence to optimum grinder pit conditions will not result in any startling improvement but there will be a change in the right direction.

Briefly, let us consider how we obtain the optimum conditions of temperature, consistency, and stone immersion.

A simple adjustment of showers will give temperatures of 185 to 190°F. in the grinder pit. Some mills may find a tendency for burning to start at these temperatures but if such is the case, I suggest they they clean their showers and rake their pits more often.

Again, by dropping the dam the required stone immersion is reached.

However, it is a little more difficult to obtain the third optimum condition, that of low pit consistency. The first thing we note is that as a result of increasing pit temperatures we have increased pit consistency and hence the gain achieved by one is partially offset by the other. The only way of decreasing our pit consistencies under these conditions is to increase shower temperatures. The economical method of doing this is to close up the paper mill and groundwood mill white water systems as much as possible, to exclude fresh water from both systems and to use excess white water in the news sulphite system. Certain mills have done a great deal in this direction in recent years and it is well worth while invest gating every possibility at your mill which will help to increase shower temperatures.

I'd like to theorize for a moment on why optimum conditions in the grinder pit tend to produce an improved groundwood pulp. Obviously no work is done on the groundwood fiber in the grinder pit. Therefore changes in grinder pit conditions are only important as they relate to changes produced in the grinding zone. The important thing to remember here is that optimum grinder pit conditions produce more favorable, but not necessarily optimum conditions in the grinding zone.

From the grinder pit a certain proportion of water and pulp is continuously carried into the grinding zone. Water is certainly a "must" in this zone but the presence of pulp brought up from the grinder pit is also desirable. This pulp then undergoes a process of further refining and beating, which we term regrinding. The presence of this pulp in the grinding zone also acts as a cushion which makes the

grinding action less intense and results in more rubbing and brushing of fibers—hence the tendency to produce a longer fibered, higher strength groundwood.

At low consistency more pulp is carried up into the grinding zone and hence the benefits as outlined, are a result. High pit temperature maintains a higher average temperature in the grinding zone which we all know to be desirable.

For optimum conditions, grinder pits should be designed with a view to elimination of dead areas and to facilitate shim removal and cleaning.

Dams should be solidly constructed so that there is no sagging.

Dams should be kept level so that there is free flow over the entire width.

Showers should be placed 4 to 6 in. from the stone and perpendicular to it. There should be adequate means of cleaning the shower so that a uniform spray is maintained across the surface of the stone.

A compliance with the above conditions will eliminate a source of nonuniform pit temperatures and nonuniform grinding zone temperatures. The results in increased stone life will pay dividends.

Fred W. Bishop (Southland Paper Mills) (*Southern Wood Species and Wood Conditions*): The accumulated years of experience by the numerous newsprint mills of the South have generally established the necessary technique for the production of quality groundwood for newsprint operation. Much of this success has been achieved by trial and error methods, but it can be explained by the basic difference between the physical dimensions of fibers from southern pine and those from spruce. The length may be the same but the southern pine fiber is two or three times as large in diameter and the magnitude of cell wall thickness is as great.

Of all the different species of pine in the South the above conditions generally hold true. In fact, to date, the grinding technique appears to be applicable to the pines of this earth so long as they are of a species that is high in pitch content.

Due to the rapid deterioration rates of southern pine, it is common knowledge by this time that successful grinding is predicated on the use of green wood or wood from underwater storage.

The conventional grinding of hardwoods in the South has been receiving very little attention with a few exceptions. Many of the gum species have been ground and they show a lot of promise but attempts to grind the oaks generally produce pulps of somewhat lower brightness. Cottonwood is presently being ground commercially with a degree of success for use in magazine paper.

The southern practices of increasing mill land ownership, reforestation, and educational programs when considered in the light of the rapid growing rate of the southern pines have decreased the local emphasis on hardwood utilization for groundwood production. This emphasis is continuing unabated for utilization in chemical and semichemical pulps.

Carleton Colbatlo (West Tacoma Newsprint Co.) (*Western Wood Species and Wood Condition*): The wood most used on the West Coast for the production of mechanical pulp is western hemlock due to its plentiful supply, followed by white fir and Sitka spruce of which there is a much lesser supply. To a smaller degree, red fir, although in plentiful supply, is used for groundwood. A small amount of cottonwood is being mechanically pulped for use in certain pulp furnishes. Included as white fir are Noble fir, grand fir, and balsam fir.

While the major supply of wood is cut during the summer and early fall, considerable logging takes place during the entire year. Many of the mills are therefore grinding wood with varying degrees of moisture, such as green wood cut less than a month, to wood that has been cut and stored in water or in a cold deck for 2 years or more. In addition, considerable amount of salvage logs consisting of previously felled, blowdown, or insect killed timber is now being used. Some of this timber is in near air-dry condition. To add to the problem the wood for these mills comes from many different types of growing sites such as the east side and the west side of the mountains, high and low elevations, wet and dry areas, and fast and slow growing sites. All of these factors affecting timber growth and condition make for a wide variation of grinding techniques necessarily practiced by groundwood mills located on the West Coast.

Data taken from the book "Pulpwoods of the United States and Canada" by Irvin H. Isenberg, wood technologist of The Institute of Paper Chemistry and published by the Institute in 1951, gives the following information using eastern white spruce for the basis of comparison:

Western hemlock reduces readily and gives a yield of 87% against 91% for white spruce. The color is only fair when compared to the excellent color of white spruce. Pulp strengths are comparable when 10 to 15% more power is used to grind the hemlock over that required for white spruce.

White fir mechanical pulp has an excellent color, gives a yield of 94%; slightly better than white spruce but again requires 15 to 20% more power to grind.

Sitka spruce gives a yield of 86%, the lowest of the woods pulped on the West Coast. The strength is good but the color is slightly creamy. The power necessary for grinding is similar to that for white spruce.

Red fir (young Douglas-fir) up to 15 in, diam. produces a good groundwood but the color is poor and the power requirements run 15 to 25% higher than that for white spruce. More power is required to make a strong pulp from red fir than any of the other West Coast softwoods.

All West Coast woods used for mechanical pulping have larger average fiber diameters and fiber lengths than eastern white spruce and hemlock. Hemlock has a diameter of 30 to 40 μ, and an average length of 4.2 mm. as compared to a diameter of 25 to 30 μ, and an average length of 3.3 mm. for white spruce. High unit pressure and grinding temperature help keep the caliper of papers containing groundwood from these woods within satisfactory limits.

In regard to grinding techniques used on the West Coast, in general, the trend is toward the use of finer grit stones, both single and multiple grit. The primary purpose in going to the finer grits is to be able to use higher unit pressures thereby increasing production as well as improving pulp strength. Pit temperatures range from 130 to 190°F. Pit consistency runs all the way from 1.5 to 4.5% A.D. Forty to one hundred pounds per square inch per unit grinding pressures are used. Stone impressions range from a cut of 6 to 12 with leads running from 1.5 in. to as high as 12 in one mill. Horsepower days per ton for grinding are in the range of from 60 to a high of 90. When possible, wood species are ground separately to facilitate better freeness control.

James C. Hunter (Powell River Co. Ltd.): (*Common Sense in Pulpstone Specifications*): The groundwood mill is a service department. The service function of the groundwood mill is to supply (in a newsprint mill) the paper machines with the quality and quantity of groundwood required at the lowest possible cost.

1. The groundwood must have the highest possible quality compatible with effect on machine efficiency and a certain maximum power and wood cost.

2. The groundwood must be at the desired freeness with minimum fluctuation.

3. The groundwood must be produced in the quantity required with each grinder at near full load to achieve a minimum of capital investment.

Other things being equal, the fulfillment of these criteria will depend on pulpstone specification choice. Over the years most groundwood mills, by trial and error, have found a standard pulpstone specification which appears adequate for their particular needs. Common sense therefore dictates that pulpstone specification choice, especially in a mill with

relatively few grindstones, must be fairly close to the specifications of the standard stone.

Specifications

Material. Material choice can be made from silicon carbide, aluminum oxide, natural sandstones, and assorted special stones. Most groundwood people who make stone specification choices have had experience with these materials. Their results with each material will determine their choice. In our experience silicon carbide has shown the best results in pulp characteristics, ease of handling, and costs. Therefore common sense dictates that the great majority of our stones will be silicon carbide.

Grit Size. Of all the decisions in determining a pulpstone specification choice of grit size is the most important. Generally increasing average grit size from the standard will result in an inherently higher freeness pulp. At the same freeness coarser stones will produce higher quality pulp but at increased power consumption. This in effect will reduce grinding capacity. If pulp quality from the standard stone is adequate and grinding capacity is limiting, common sense dictates that a substantially coarser grit size than standard should not be chosen. Grit size choice then will be determined by the original criteria.

Grade. Wear rates (cubic inches of stone per moisture-free ton) of "harder" stones are lower than "softer" stones and therefore use of "harder" stones should result in lower stone costs. The limitation however is ease of burr application and surface pattern control. Common sense dictates that the stone should be "hard" enough to minimize stone costs without causing excessive burring trouble.

Structure. Change of density of grain spacing should have an effect similar to changing average grit size. This choice then is dependent on grit size choice and the combined effect on the original criteria.

Bonding. Changes in bond say from the standard vitreous (V) bond to a more resistant bond must be considered with the "hardness" rating choice. The combined effect on burr application and surface pattern control will determine the success of the bond choice.

In mills with a large number of stones, the great majority of standard specifications, there should be the odd stone with specifications slightly different from the standard. This will allow assessment of the various stone characteristics under mill conditions and so lead to improvements in specification choice.

Coarse Grinding Followed by Refining

F. S. HOHOLIK

In conjunction with plans for a speed up of the paper machine, the need for approximately 35 tons per day of additional groundwood production became apparent. After consideration of various alternatives for providing more firm groundwood capacity, the possibility of grinding coarse with high production per grinder, followed by finishing the pulp in double disk refiners, offered a low capital cost means of achieving this objective.

A sample of groundwood having a Canadian Standard freeness of 350 ml. was produced on one of our regular grinders with a power input of approximately 35 hp. days per air-dry ton. This sample was submitted to the Fiber Products Laboratory in Springfield, Ohio, where it was refined in a double disk refiner to a pulp which we considered to be of acceptable news grade. For data see Table II, Samples No. 62 and 63. Although this pulp did not show the superior tear usually associated with refiner produced groundwood,

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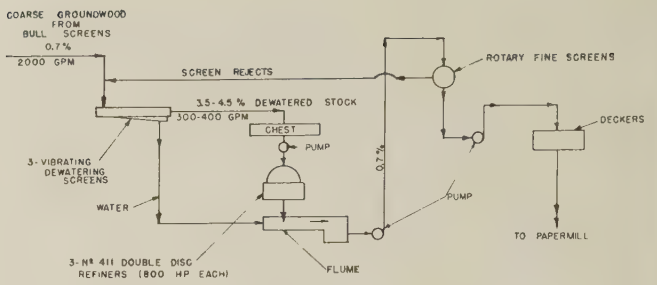

Fig. 1. Flow sheet—coarse groundwood refining system, Manistique Pulp & Paper Co.

this was considered to be due to the fact that the fine grit stones were not optimum for production of the coarse groundwood for refining.

It has been later reported (*1*) that a mill-produced coarse groundwood using a coarser grit stone better adapted for this purpose could be satisfactorily refined to a finished pulp having equal or better strength attributes as compared to pulp produced completely on grinders with equal power input.

On the basis of the above experimental results, it was decided to proceed with the refiner installation as described below.

DESCRIPTION OF GRINDER AND REFINER SYSTEM

The mill of the Manistique Pulp & Paper Co. is a one-machine mill producing grades ranging from novel news to standard news, directory and groundwood printing papers.

The general flow sheet of the groundwood mill and the newly installed refiner system is shown in Fig. 1.

There are six Voith magazine grinders equipped with water wheels capable of delivering 900 hp. each under normal water conditions. Three of these grinders are also equipped with electric motors of 1250, 1250, and 1500 hp., respectively.

Under average water conditions, the groundwood mill has a capacity of 55 to 65 tons per day, depending on groundwood grade demanded for various grades on the paper machine.

The stones are 60-in. diam. by 54 in. face. All grinders except one are equipped with standard news stones having an average grit size of 60.

One grinder only has been equipped with a special Norton stone, A303-N7VG, having an average grit of 30. This stone was selected to provide a long free pulp, free of chop and having the requisite strength potential for maximum strength development in the subsequent refining process. This stone has normally been sharpened with a 10 cut spiral burr, $1^1/_2$ in. lead for novel news grade and 14 cut burr, $1^1/_4$ in. lead for news grade groundwood. A similar procedure is used on the fine grit stones when operating on coarse groundwood.

As the finer grit stones require replacement, it is anticipated that coarser stones of the type described above will be installed.

The combined grinder pit stock, approximately 7% consistency as leaving the grinders, flows to two bull screens having $^3/_8$ in. round perforations. The bull screen accepts at approximately 0.7% consistency are pumped through three Allis-Chalmers single deck, low head vibrating screens for thickening. These screens are each 9 ft. long by 5 ft. wide and have graduated perforations of 0.032, 0.041, and 0.069 in. enumerated from the freed end.

The "rejects" or coarse fraction to be refined come off these screens at 3.5 to 4.5% consistency and drop into an agitated chest below.

The accepts or "white water," having consistency of 0.3 to 0.4%, by-pass the refiners and recombine with the refined product later.

Although the vibrating screens were installed primarily for the purpose of thickening the bull screen accepts ahead of the refiners, it has proved that they have a major classifying

Table I. Operating Data

Day no. (1)	Paper grade produced (2)	Groundwood production air-dry tons (3)	Production per grinder day, air-dry tons, (4)	Grinders			Refiners				
				Brake hp. days (5)	Brake hp. days/air-dry ton (6)	% Total power (grinders) (7)	Brake hp. days (8)	Brake.hp days/air-dry tons (9)	% Total power (refiners) (10)	Total brake hp. days/air-dry tons (11)	% Total Groundwood refiners (12)
1	Novel news	44.37	22.19	1559	35.14	59.6	1057	23.82	40.4	58.96	35.4
2 (22 hr.)	"	32.71 (24 hr.)	27.31	917	28.03	47.6	1010	30.88	52.4	58.91	44.0
2A (2 hr.)	...	...	...	...	...	...	...	...	...	...	49.1
3	"	37.48	30.24	982	26.20	52.1	903	24.09	47.9	50.29	56.1
4	Std. news	42.14	20.64	1754	41.62	61.9	1081	25.65	38.1	67.27	60.2
5	"	40.23	20.12	1705	42.38	60.4	1117	27.77	39.6	70.15	48.3
6											
6 hr.	"	10.38	16.65	348	33.53	54.0	297	28.61	46.0	62.14	...

[a] On day no. 2, 2 grinder No. (coarse stone) was isolated for 2 hr. and this period has been designated in Tables I and II as 2A.
[b] Refiner hp. days/air-dry ton (column 9) is calculated on total pulp production.
[c] Percentage of total pulp through refiners, column 12, calculated from consistencies of feed, accepts, and rejects at vibrating screens.

or selective action and roughly 50% of the total pulp passes through and is not further refined. The percentage degree of this separation is shown in Table I.

There is some probability that the by-passing of this relatively large fraction of pulp around the refiners may result in a net power saving, although data to determine this point have not been established to date. While this accept material has sufficient fineness for use on the paper machines, its strength is somewhat inferior either to the refiner product or to the subsequent recombined finished pulp.

On the other hand, the preliminary experimental refining work referred to in the introduction was carried out on the total bull screen accepts and it is possible that the white water fraction is capable of further strength development if refined further as a total stock.

This could be accomplished if a decker rather than the vibrating screens were used for thickening ahead of the refiners.

A decker would also permit higher consistency to the refiners.

Rejects from the vibrating screens are pumped to three Bauer 40 in. double revolving disk refiners at 3.5 to 4.5% consistency. These refiners each have two 400-hp. motors installed making a total of 2400 installed hp. on the averaged equal to or slightly less than that applied on the total pulp at the grinders.

The refiner product recombines with the by-passed white water and the composite stock is rotary fine screened and thickened on deckers in the conventional manner.

The rotary fine screen rejects are returned to the bull screen pit and from there are pumped with the bull screen accepts to the double disk refiners for reduction into suitable mechanical pulp. In this respect, the coarse grinding-refining equipment is required.

The deckered finished groundwood is combined with long fibered pulp and other furnish components. The mixed stock is cleaned in a Bauer 12 in. cleaner system-prior to the paper machine headbox.

DISCUSSION OF OPERATING RESULTS

Production and power data are given in Table I. During the test period when these data were derived, the mill demand for groundwood was less than the 90 ton per day design capacity of the grinder-refining system. Therefore, neither all the grinders nor all the refiner units were operated throughout most of this period. However, we attempted to maintain the ratio of approximately 60% of total power requirement on the grinders and 40% on the refiners, since this is the system design.

Reference is made to Table II for pulp quality data. It should be noted here that all pulp tests were taken prior to fine screening.

Wood species normally used consist of about 70% spruce and balsam, balance being jack pine and poplar.

Data have been derived for three days (No. 1, 2, 3) operation on the relatively high freeness "novel news" grade mechanical pulp and three days (No. 4, 5, 6) operation on standard news grade mechanical pulp.

For each of the six days, pulp samples were taken every 2 hr. at the locations designated in Table II. An 8-hr. composite of four samples from each point was made up for testing.

The production and power figures shown in Table I for each day are on a 24-hr. basis.

Since separate watt-hour readings were not available from individual grinders or refiners, power figures are shown only for total grinders or refiners operating. Likewise, it was not possible to determine throughput at individual grinders or refiners. However, an effort was made to distribute both power and throughput equally to the individual units, with the exception that No. 2 grinder with the coarse stone normally handled more tonnage than the other grinders.

Operation on Novel News Grade

Total power input (grinders plus refiners) was in the range of 50–59 brake hp.-days per air-dry ton. From 47.6 to 59.6% of the total power input was applied at the grinders, balance at the refiners. Pulp output per stone ranged from 22.19 to 30.24 air-dry tons per 24 hr. Normal pulp output on this grade is approximately 17 air-dry tons per stone on water power.

The finished pulp (samples 9, 17, 23, 32) is considered of satisfactory quality for novel news. Burst and breaking length are about standard and the tear is slightly higher as compared pulp normally produced completely on the stones at equal power input.

The accepted pulp passing through the vibrating screen is low in both freeness and strength. The refiner output pulp has strength values superior to conventional groundwood of this grade. However, this refiner fraction has received more power input per ton since only approximately half the total pulp passes through the refiners.

The recombined fractions have strength factors below their weighted arithmetical average but, as stated above, are adequate for this grade.

Production from the special coarse stone in No. 2 grinder was isolated for a 2-hr. period (designated in Table II as 2A).

Pit sample No. 18, at a comparatively high freeness of 479 ml., had very low strength. The refiner product (sample 22), however, had the highest strength shown in the novel news grade data. The recombined pulp was about equivalent in strength to the other samples shown in this series.

During the period of novel news grade operation, No. 1 Refiner had A-400-DE plates, No. 2 had No. A-404 plates,

Table II. Pulp Quality Data

Sample No.	Consistency, %	Tappi Standard: Can. Std. freeness, ml.	Tappi Standard: Burst factor	Tappi Standard: Tear factor	Tappi Standard: Breaking length, m.	Bulk, cc./g.	G.E. brightness, %	Opacity, %
Day No.—1 Novel News Grade								
1. No. 2 Grinder pit	8.31	326	3.36	36.1	993	3.37	55.0	95.5
2. No. 3 Grinder pit	7.10	274	3.24	33.4	1000	3.41	57.0	95.2
3. Feed to vibrating screen	0.61	207	6.88	49.0	1610	2.89	56.0	97.0
4. Vibrating screen rejects	3.70	496	4.74	45.6	1141	3.44	56.5	92.3
5. Vibrating screen accepts	0.41	108	6.91	34.7	1810	2.54	56.1	98.5
6. No. 1 Refiner output	...	187	12.8	60.2	2860	2.60	58.0	96.0
7. No. 2 Refiner output	4.38	78	16.5	55.7	3610	2.30	58.8	96.9
8. No. 3 Refiner output	3.90	157	11.8	58.1	2520	2.66	58.8	95.5
9. Recombined pulp	0.71	108	9.98	39.1	2315	2.45	56.0	98.1
Day No. 2—Novel News Grade								
10. No. 2 Grinder pit	7.70	395	3.14	31.1	1080	3.34	55.2	94.5
11. No. 3 Grinder pit	7.15	306	3.44	34.5	928	3.29	56.5	95.6
12. Feed to vibrating screens	0.75	249	5.32	38.3	1440	3.00	54.3	96.0
13. Vibrating screen rejects	3.88	620	3.55	44.0	968	3.46	54.0	92.0
14. Vibrating screen accepts	0.46	97	5.50	31.1	1450	2.37	58.0	99.0
15. No. 1 Refiner output	3.85	351	8.69	49.7	2000	2.77	56.3	95.0
16. No. 2 Refiner output	4.40	233	10.5	50.3	2090	2.80	57.5	96.3
17. Recombined pulp	0.62	154	6.25	36.0	1620	2.80	57.1	97.6
Period No. 2A—Novel News Grade								
18. No. 2 Grinder pit only	...	479	1.85	25.9	684	3.90	53.5	93.2
19. Feed to vibrating screens	0.62	206	7.06	59.7	1670	2.79	53.0	96.8
20. Vibrating screen rejects	4.42	674	5.73	72.6	1150	3.43	55.0	92.1
21. Vibrating screen accepts	0.34	84	7.15	36.7	1770	2.34	52.5	99.5
22. No. 2 Refiner output	...	198	19.2	74.4	3830	2.41	56.5	97.0
23. Recombined pulp	0.40	86	9.36	46.6	2300	2.40	54.0	98.7
Day No. 3—Novel News Grade								
24. No. 1 Grinder pit	...	349	3.67	33.1	990	3.31	58.0	94.2
25. No. 2 Grinder pit	...	487	2.96	35.9	864	3.65	55.9	94.0
26. Feed to vibrating screens	0.60	265	7.47	55.4	1510	2.87	54.2	96.5
27. Vibrating screen rejects	3.64	636	7.37	73.7	1360	3.21	55.0	91.3
28. Vibrating screen accepts	0.31	105	7.86	40.2	2110	2.47	54.9	98.0
29. No. 1 Refiner output	...	280	15.5	68.0	3200	2.46	55.6	94.8
30. No. 2 Refiner output	...	59	14.7	44.5	3320	2.29	53.2	98.5
31. No. 3 refiner output	...	162	14.3	43.3	3490	2.31	55.0	98.0
32. Recombined pulp	0.49	140	10.4	46.4	2380	2.39	55.7	97.5
Day No. 4—Standard News Grade								
33. No. 1 Grinder pit	7.27	369	3.74	33.0	855	3.34	57.0	94.0
34. No. 2 Grinder pit	6.67	306	5.97	42.5	1360	3.20	57.6	95.0
35. Feed to vibrating screens	0.64	236	8.39	52.9	1720	2.84	56.8	97.0
36. Vibrating screen rejects	4.33	546	6.85	64.2	1691	3.32	57.0	92.0
37. Vibrating screen accepts	0.28	88	9.64	37.3	2330	2.38	56.2	98.5
38. No. 1 Refiner output	5.37	349	9.63	52.0	2080	2.97	57.0	94.5
39. No. 2 Refiner output	6.73	162	16.5	58.3	3870	2.44	58.2	91.5
40. No. 3 Refiner output	1.06	175	15.6	60.6	3390	2.59	58.0	96.5
41. Recombined pulp	0.96	149	10.7	60.0	2760	2.78	57.0	96.0
Day No. 5—Standard News Grade								
42. No. 1 Grinder pit	7.13	326	4.52	34.6	774	3.31	56.0	95.0
43. No. 2 Grinder pit	8.15	306	4.63	40.3	1230	3.31	58.0	95.5
44. Feed to vibrating screens	0.57	250	5.70	42.2	1310	3.10	56.5	96.5
45. Vibrating screen rejects	4.87	620	4.03	46.3	945	3.54	56.0	92.0
46. Vibrating screen accepts	0.32	73	7.71	32.0	1765	2.43	55.0	97.2
47. No. 1 Refiner output	4.72	166	11.7	47.2	2700	2.64	58.0	97.1
48. No. 2 Refiner output	4.74	140	14.5	58.2	3130	2.54	58.2	97.0
49. No. 3 Refiner output	4.52	221	10.6	52.6	2440	2.68	57.5	96.0
50. Recombined pulp	0.60	125	10.2	42.0	2350	2.49	57.0	97.5
Day No. 6—Standard News Grade								
51. No. 1 Grinder pit	...	330	4.62	34.6	1240	3.16	57.0	94.5
52. No. 2 Grinder pit	...	255	8.60	55.0	2100	3.04	58.5	95.0
53. Feed to vibrating screens	...	202	7.96	49.7	1970	2.64	57.0	96.5
54. Vibrating screen rejects	...	620	5.84	69.4	1540	3.38	57.2	92.0
55. Vibrating screen accepts	...	75	8.04	37.4	1930	2.33	57.2	98.0
56. No. 1 Refiner output	...	165	18.1	71.3	3750	2.42	57.0	94.0
57. No. 2 Refiner output	...	198	19.7	74.8	3980	2.27	59.0	96.5
58. No. 3 Refiner output	...	182	15.7	66.4	3300	2.50	58.0	96.5
59. Recombined pulp	...	103	11.2	45.8	2300	2.39	58.0	99.0
Regular Novel News Grade								
60. Grinder pits (all grinder pulp)	...	164	7.60	39.8	1940	3.00	57.0	97.3
Regular News Grade								
61. Grinder pits (all grinder pulp)	...	110	10.2	44.7	2840	2.40	57.3	98.4
Fiber Products Laboratory Trial								
62. Coarse groundwood supplied for Fiber Products Lab. Test (approx. 35 hp. days/air-dry ton)	...	350	4.50	29.2	1300	3.10	53.5	96.8
63. Sample 61 laboratory refined (No. 400 Refiner, 28.9 hp days/air-dry ton)	6.02	125	12.2	31.3	3050	2.30	51.7	98.8

Table III. Fiber Classification Data

Sample No.	On 28 Mesh	On 48 Mesh	On 100 Mesh	On 200 Mesh	Through 200 Mesh
			%		
		Day No. 3—Novel News Grade			
24. No. 1 Grinder pit	24.73	22.32	16.88	12.35	23.72
	24.73	47.05	63.93	76.28	100.00
25. No. 2 Grinder pit	29.61	18.49	12.68	10.38	28.84
	29.61	48.10	60.78	71.16	100.00
26. Feed to vibrating screens	27.96	20.02	14.14	11.80	26.08
	27.96	47.98	62.12	73.92	100.00
27. Vibrating screen rejects	51.38	14.52	10.69	7.38	16.03
	51.38	65.90	76.59	83.97	100.00
28. Vibrating screen accepts	9.55	14.63	13.24	12.17	50.41
	9.55	24.18	37.42	49.59	100.00
29. No. 1 Refiner output	31.93	14.96	11.79	12.29	29.03
	31.93	46.89	58.68	70.97	100.00
30. No. 2 Refiner output	3.94	13.21	20.16	24.97	37.72
	3.94	17.15	37.31	62.28	100.00
31. No. 3 Refiner output	12.11	20.47	19.16	19.53	28.73
	12.11	32.58	51.74	71.27	100.00
32. Recombined pulp	15.53	16.49	12.52	12.47	42.99
	15.53	32.02	44.54	57.01	100.00
		Day No. 6—Standard News Grade			
51. No. 1 Grinder pit	19.84	22.59	17.56	12.91	27.10
	19.84	42.43	59.99	72.90	100.00
52. No. 2 Grinder pit	27.81	19.57	13.25	10.23	29.14
	27.81	47.38	60.63	70.84	100.00
53. Feed to vibrating screens	20.89	16.43	12.30	9.84	40.54
	20.89	37.32	49.62	59.46	100.00
54. Vibrating screen rejects	38.32	18.61	10.80	7.69	24.58
	38.32	56.93	67.73	75.42	100.00
55. Vibrating screen accepts	7.70	13.06	12.86	11.61	54.77
	7.70	20.76	33.62	45.23	100.00
56. No. 1 Refiner output	23.24	20.03	15.22	13.88	27.63
	23.24	43.27	58.49	72.37	100.00
57. No. 2 Refiner output	27.06	19.34	14.13	12.50	26.97
	27.06	46.40	60.53	73.03	100.00
58. No. 3 Refiner output	14.22	17.21	13.20	9.77	45.60
	14.22	31.43	44.63	54.40	100.00
59. Recombined pulp	13.78	16.39	13.00	10.56	46.27
	13.78	30.17	43.17	53.73	100.00
60. Regular novel news-grinder pit (all grinder produced)	12.23	16.92	15.10	19.11	36.64
	12.23	29.15	44.25	63.36	100.00

and No. 3 had No. A-406 plates installed. The only consistent difference in pulp quality apparent is the lower freeness from the finer tooth A-404 plates in No. 2 refiner. These plate patterns are illustrated in Fig. 3.

Standard News Grade Operation

It is noted from Table I that the total power input for this grade was higher than the freer novel news grade. Total power (grinders plus refiners) varied from 62.14 to 70.15 brake hp. days per air-dry ton. Percentage of total power used on the grinders was also higher, ranging from 54.0 to 61.0%.

Examination of pulp data indicates that No. 2 coarse stone produced consistently higher tear at the grinder pit as compared to the regular 60 grit stones.

With the somewhat higher total power input for this grade and the lower freeness of the pit stock, pulp from the refiners showed excellent burst, tear, and breaking length.

Tear especially was well maintained in the re-combined fractions (samples 41, 50, 59) and these finished pulps are considered equivalent or slightly superior to conventional groundwood of this grade at equal power input.

A-400-DE plates were used in all refiners on the standard news grade operation.

Fiber Classification Data

Fiber classifications were run using a Bauer-McNett fiber classifier on all samples.

Classification data for days No. 3 and 6 are shown in Table III.

The selective effect of the vibrating screens in the mill system is quite apparent with a concentration of fines and a large reduction of the coarse and fraction in the accepts from these screens.

There is also a corresponding decrease in fines and an increase in coarse fraction apparent in the rejects from the vibrating screens (refiner feed).

The recombined finished pulp has a fiber distribution generally comparable to the conventional all grinder pulp produced at this mill as shown in Table III, sample 60.

CONCLUSION

In conclusion it may be stated that the coarse grinding followed by refining technique will produce mechanical pulp of equivalent or better quality than pulp produced completely on grinders at the same total power input.

It has also proved possible to double the production per stone at half the normal power input and finish of the resultant coarse groundwood to an acceptable pulp in the double disk refiners. This will permit an existing groundwood mill to double its production without installing additional grinders.

Flexibility for immediate adjustment of pulp freeness and quality is provided by the refiners.

While the data presented do not necessarily represent the ultimate results that may obtained, all grades of pulp produced by the described system have run well on the paper machine with no adverse affect on machine running conditions or paper quality.

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New Wood Handling System and Groundwood Mill at Powell River

JAMES C. HUNTER

On April 21, 1955, the Powell River Co. announced its intention to install another newsprint machine at Powell River. Besides the machine additional power and a new groundwood mill were required. Previous to the announcement arrangements were made with the B. C. Electric Co. to furnish the required power via a high tension line from the Lower Mainland.

Following the announcement the Technical Services Department and operating personnel collaborated on process flow design and preliminary equipment choice for the proposed new groundwood mill. These data were forwarded to the consultants, Sandwell and Co., to serve as the basic design for the new mill.

Long-range plans for the renovation of the groundwood mills at Powell River had been active for some time. In line with these plans Koering-Waterous Co. were commissioned by the Powell River Co. to build two 64-in. Great Northern grinders (basically the same as the standard 48-in. model) capable of producing 60 ovendry tons per grinder day.

The two prototype grinders were installed in GP-2 groundwood mill in 1952. Each grinder is driven by a 4500 hp. 50 c.p.s. synchronous motor and, with a partition in the magazine, grinds 32-in. blocks from the normal wood supply. The performance of the grinders met all expectations. Accordingly, the choice of grinders for the new groundwood mill, or GP-4 as it was designated, was four 64-in. Waterous Great Northern grinders.

The new groundwood mill building was designed to house a unit of eight grinders and processing equipment. The project was completed and groundwood production started in January, 1956.

THE GP-4 GROUNDWOOD MILL AT POWELL RIVER

A. Wood Supply

Wood demand of about 110 M BCLS per day can be obtained from three sources or combination of these sources (Fig. 1):

(1) Salvage bundle booms

(2) Small flat-boom logs up to 22-in. diam.

(3) Sawn wood from the sawmill. This source will be used only during emergencies and will be obtained by raising every second saw on a slasher deck cutting 32-in length blocks for the 3-pocket grinders.

While salvage wood is intended to be the major source of GP-4 wood supply, if barking capacity becomes a limiting factor and/or decreased use of salvage wood is indicated, there will be about 210 M BCLS per day of small flat-boom logs available for GP-4 via the ring barker.

Species control is obtained by programming booms to the whole mill in the desired species percentages. Because all groundwood produced eventually is mixed species, control is maintained within a fairly narrow range.

James C. Hunter, Powell River Co. Ltd., Powell River, B. C.

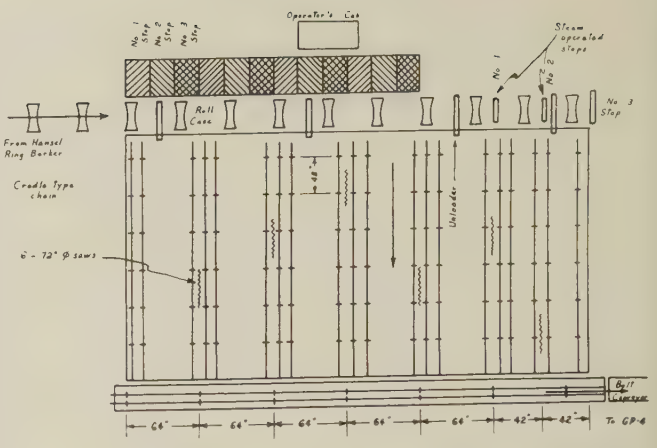

Fig. 2

B. Wood Preparation

(1) Salvage bundles averaging about 3 M BCLS each are raised by a 40-ton crane to a break-up deck.

(2) Flat-boom logs are removed from the water by a standard inclined log haul.

Logs are cut to length by a 108-in. diam. cut-off saw (maximum 26 ft. 8 in.). The first cut and second if required

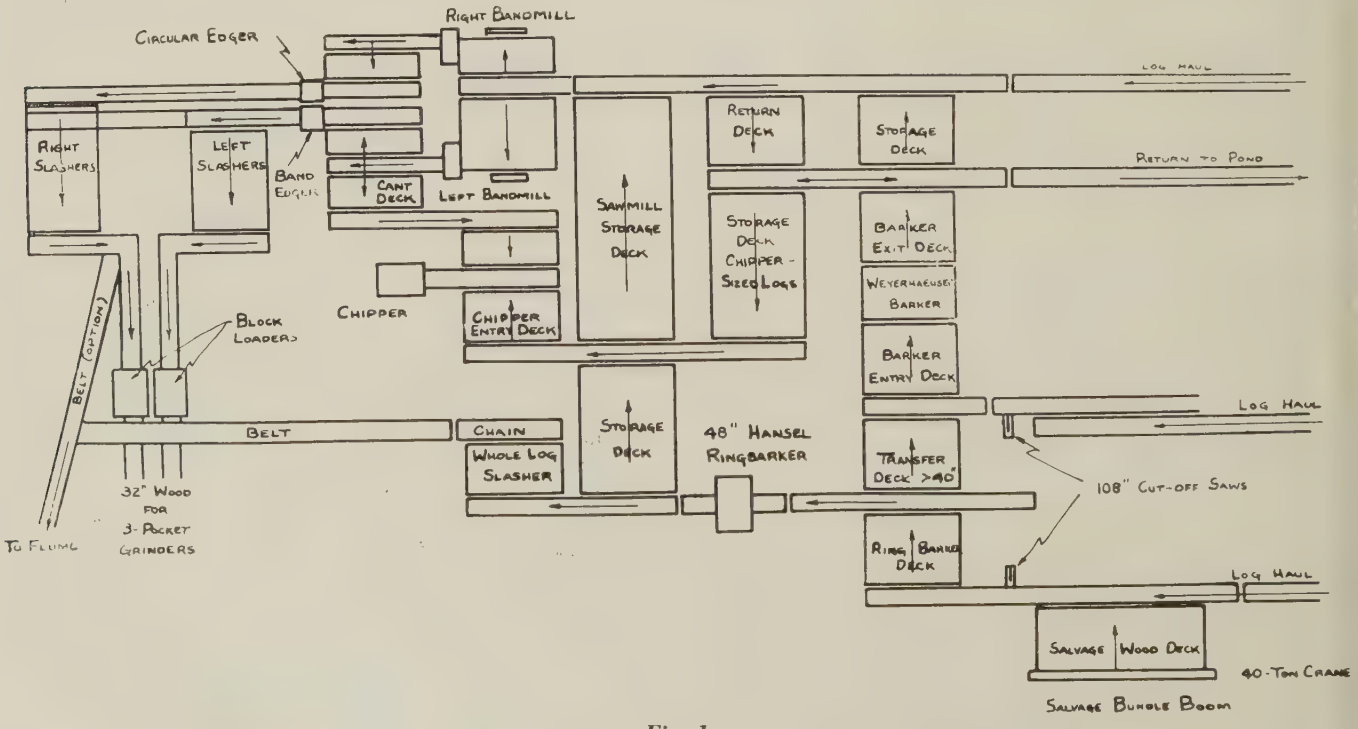

Fig. 1

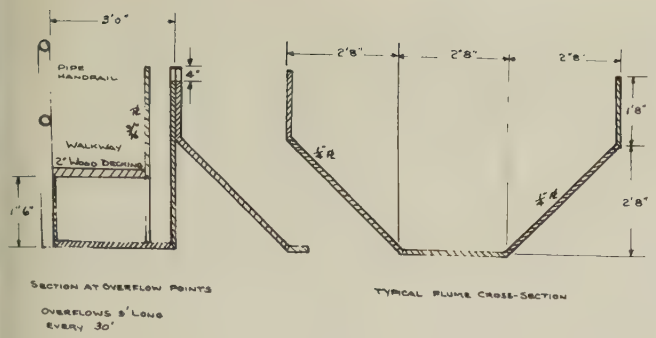

Fig. 3

provides 64-in. multiples. The pieces are barked on a 48-in. Haneel ring barker. Pieces of suitable diameter and length are run to the whole log slasher as required, excess and large diameter pieces are detoured to the chipper and sawmill.

Bundles which contain fir logs must be used on the whole log slasher to prevent fir entry into the sulfite chips. To utilize fully the second or third piece (random lengths) the roll case at the whole log slasher (Fig. 2) is provided with two steam operated stops which, in conjunction with the saw setup will provide 42-in. and longer blocks from any usual piece length. With the first stop up the operator notes the position of the trailing end of the piece relative to an indicating board and places the forward end of the piece against one or other of the three stops as required.

Whole Log Slasher Data
Maximum log diameter—22 in.
No. of saws—6
Saw diameter—72 in.
Teeth—Detachable
Peripheral speed—11,000 f.p.m.
Drive—40 hp. each
Chain speed—30 f.p.m.
Chain type—Cradle arm
Cradle spacing—48 in.

C. *Block Transport*

Blocks from the slasher fall onto two S.S. 40 chains slightly longer than the width of the slasher deck, thence to a 240-ft. horizontal belt. Leaving the horizontal belt, the blocks are chuted onto a similarly inclined belt, 330 ft. long, for transport over the main roadway and mill buildings. This belt extends back to the emergency sawn-wood entry from the saw mill. Both belts are 48-in. wide in standard roller supports.

From the inclined belt the blocks go down a chute to a flume 1200 ft. in length. The flume passes through the old mill area and adequate provision for overflows had to be included in the flume design (Fig. 3). Overflows, when they occur, are diverted into a duct alongside the flume. The top to the duct serves as the walkway alongside the flume. Flume sluicing water, about 3000–5000 Imp. g.p.m., is

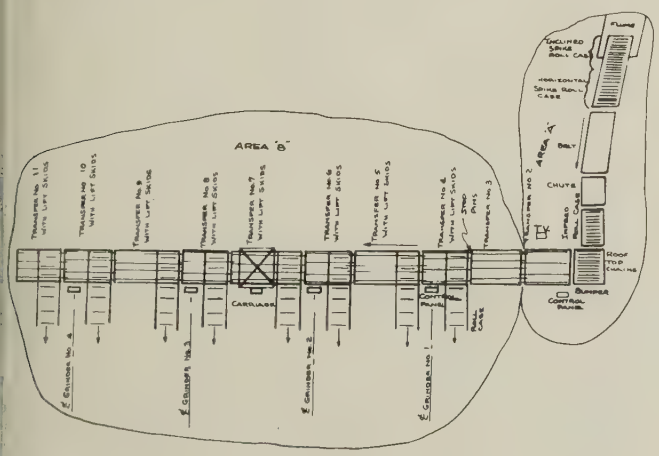

Fig. 4

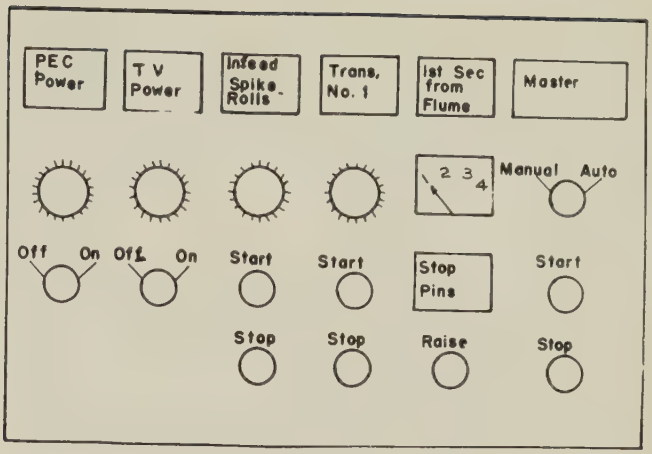

Fig. 5

piped from no. 2 penstock nearby. The water is discharged to sewer at the flume exit. Flume level control is obtained by the use of removable dam boards at the exit. The flume provides 8 hr. storage for GP-4.

D. *GP-4 Block Handling System*

The GP-4 block handling system is divided into two areas.

1. *Area A.* This area is controlled by the flume operator stationed at the Area A control panel (Fig. 5) near the right-angle turnpoint. Facing the operator is a closed-circuit T.V. set showing the exit end of the flume and the inclined and horizontal spike roll cases.

Procedure for bringing blocks into the grinder room is as follows:

(a) The operator starts each conveyor section in sequence but not including the partially submerged inclined roll case.

(b) While observing the T.V. picture the operator starts and stops the inclined section to allow single blocks only to run on the horizontal roll case section and through the conveyor system to the transfer deck.

2. *Area B.* Loading of all four grinders and storage of blocks on the transfer decks is done by the loader operator

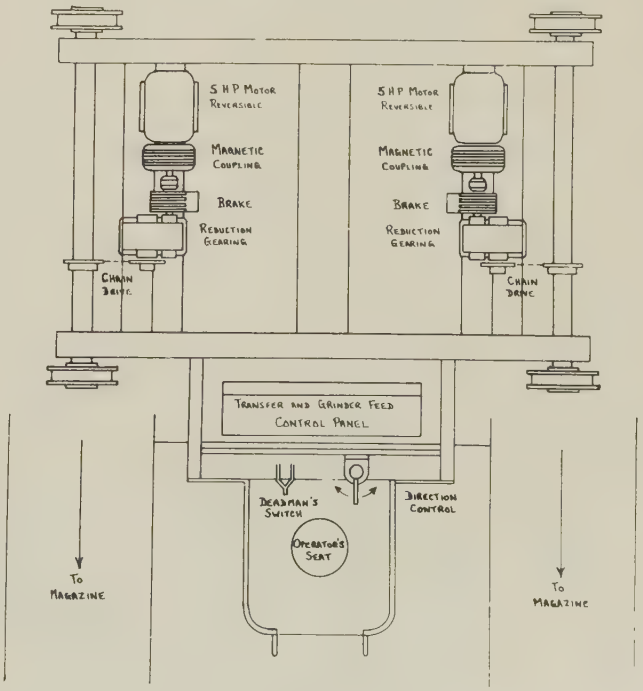

Fig. 6

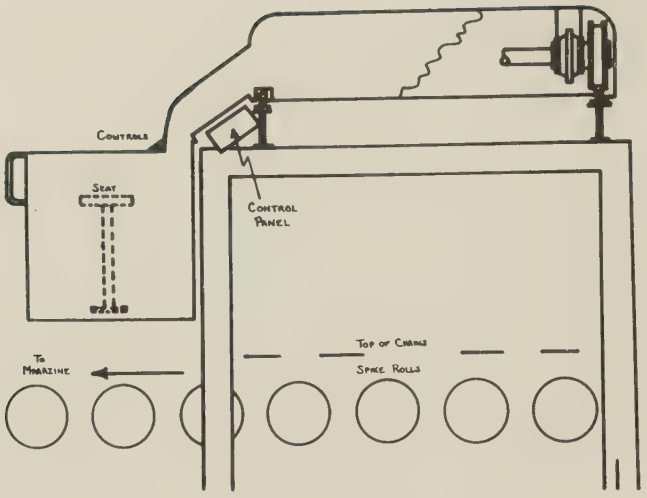

Fig. 7

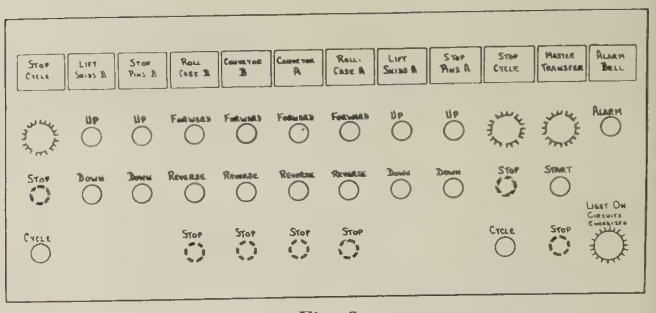

Fig. 8

stationed in a rail carriage (Figs. 6 and 7) which runs above and along the transfer deck sections. Control panels (Fig. 8) for block transfer and grinder loading are located at the center-line of each grinder. When a grinder pocket is filled, reloading of the magazine is accomplished as follows:

(a) The operator in the carriage proceeds to the grinder and centers the carriage within 3 in. of the grinder center-line. A limit switch is depressed by the carriage, which activates a green light on the control panel and also overrides the circuits controlling the stop-pins, lift skids, and roll cases for both pockets of the grinder.

(b) With the area above the roll case clear the lift skid is dropped and the roll case started.

(c) Single blocks are dropped on the roll case by movement of the transfer chain leading to the roll case and intermittent use of the stop pins.

(d) Moving the carriage from the center-line of the grinder restores the stop-pins to normal, stops the roll case, raises the lift skids, and turns off the green light.

Stop-cycle, hand controls are also located on the grinder walkways for use during jams.

E. Grinders

The four 64-in. Waterous Great Northern grinders are each powered by a 5000 hp. 60 c.p.s. synchronous motor. Pocket loading and grinding is done automatically through limit switches and valve sequence operation as on the standard 48-in. Waterous model. Load can be controlled by the individual grinder governors or by a master governor acting through the individual governors, which maintains desired B. C. electric power with the major portion to GP-4 and a small amount channelled through a 60 to 50 c.p.s. converter to the mill loop. B. C. electric power is maintained constant and variations in demand are taken up by the grinder motors.

The grinders are equipped with pit temperature control on the rear shower and manual control on the top shower and operating equalibrium conditions are as follows.

Pit temperature (controlled), 180°F.
Shower temperature (controlled), 130°F.
Pit consistency, 2.0%.
Average load, 3500 kw.

The Norton artificial grinder stones are 67 in. in diam. and 69 in. in length. Stone compositions are:

Grinder No. 1—37C60 Special—P7V av. 70 grit—$^1/_2$ 60, $^1/_2$ 80
Grinder No. 2—37C701 P7V
Grinder No. 3—37C607 P7V $^1/_2$ 60, $^1/_4$ 80, $^1/_4$ 90
Grinder No. 4—37C602 P7V $^1/_2$ 60, $^1/_4$ 70, $^1/_4$ 80

F. Screen Room

The screen room is designed for brightening after the deckers to achieve:

1. High grinder shower temperature
2. Optimum brightening conditions

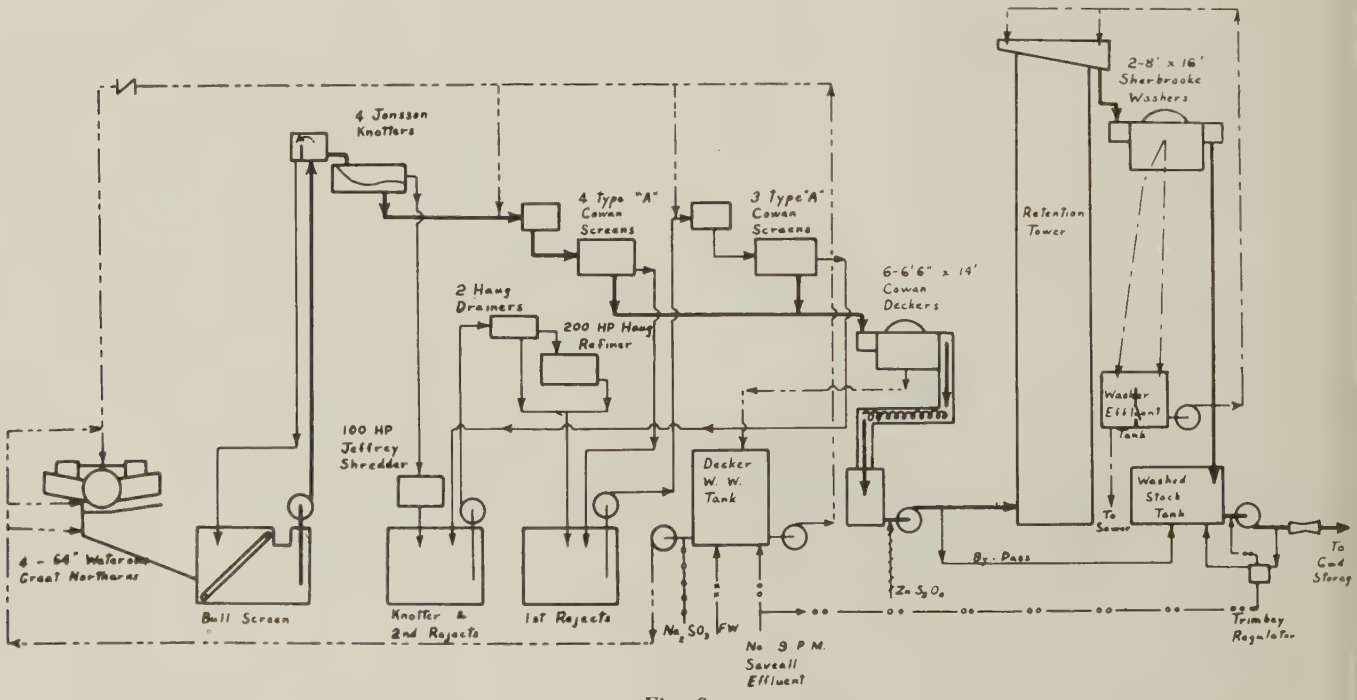

Fig. 9

3. Minimum corrosion by exclusion of zinc hydrosulphite from the grinder and screen rooms.

(a) The bull screens are standard inclined raker screens with 1-in. diam. perforations in high-chrome steel plates. Provision has been allowed for reclamation of bull screen rejects via the Jeffery shredder but as yet this is not in use.

(b) Bull screen accepts are pumed to four Jonsson knotters with $^3/_{16}$-in. diam. perforations in 316 S.S. plates.

(c) Knotter accepts are screened in seven type A Cowan screens. All parts in contact with the stock are either 3.6 S.S. or rubber-clad. Screen plates are 18 gage 316 S.S. with 0.07-in. diam. perforations. Headbox arrangements permit the use of four firsts and three seconds or five firsts and two seconds.

(d) Screen accepts are dewatered in six 6 ft. 6 in. by 14 ft. Cowan single end discharge gravity deckers. The inner shell is constructed of $^3/_{16}$ in. with $^3/_8$ in perforations. Backing wire on the shell is 10 mesh and facing wire is 30 or 40 mesh. All parts in contact with the stock are 316 S.S. Stock is doctored from the deckers by rubber-covered couch rolls equipped with balance arms.

At 0.55 to 0.60% fed consistency capacity of the deckers is about 40–50 o.d. tons per day with discharge consistencies of about 4 to 5% o.d.

(e) Refining System. Knotter rejects pass through a 100 hp. Jeffery shredder, join second screen rejects, and are pumped to two Haug drainers, dewatered to 12% o.d. and thence to a 200 hp. eight-roll Haug refiner. With normal operating conditions no rejects losses occur with the refiner at 70 to 80% full load.

G. Brightening System

(a) Zinc hydrosulphite is added to the suction of the deckered stock pump to the bottom of the retention tower. The tower is 16 i.d. by 70 ft. high and constructed of pressure creosoted wood staves. At 4.5% consistency retention time is about two hours. Stock temperature is 140°F.–150°F. Stock is sluiced from the overflow ring of the tower by recirculated washer effluent.

(b) The brightened stock is washed in two 8 by 16 ft. long Sherbrooke 316 S.S. washers. Stock removal is effected by water-jet doctor dischargers. Washed stock flows by gravity to the washed stock tank.

H. Washed Stock Pumping System

To minimize freeness and pulp quality variations washed stock from GP-4 is pumped 1500 ft. to the central mill blending tanks to be mixed with groundwood from the other mills and the mixture distributed to the various paper machines.

I. Materials of Construction

All tanks and stock flumes are tile lined. Pressure creosoted wood stave pipe and 316 S.S. stainless fittings are installed throughout the mill. All equipment, as far as possible, is of 316 S.S. construction.

J. Production and Pulp Quality

During the first two weeks in October average GP-4 production and pulp quality statistics were:

1. Pit freeness—92 C.s.F.
2. Production—60 o.d. tons per grinder-day
3. Load—90% full load
4. Power consumption—1420 kw.-hr. o.d. tons
5. Brightness—58.0 G.E. units
6. Pulp quality at 85 C.S.f. (Deckered stock)
   a. % Mullen—36.5
   b. Tear factor—51.0

K. Personnel

A GP-4 shift crew consists of:
1. Jiggerman.
2. Screen-tender.
3. Loader operator.
4. Flume operator.
5. Spare.

Present man-power production rate is about 2.0 o.d. tons per man-hour. The addition of another four grinders in the future will require at most another two men and man-power production rate will increase to about 2.0 a.d. tons per man-hour.

Panel Discussion: Tailoring Groundwood for End Use

S. R. Parsons (Consolidated Water Power & Paper Co.) (*Manufacture of Groundwood for Use in Book Paper*): In producing a coated groundwood sheet suitable for top quality halftone printing the primary requirements are for a groundwood of high brightness and freedom from dirt and shives. High bulk, high opacity, good absorbency for printing inks, and good running qualities on the paper machine are also essential.

Our grinders are largely pocket grinders with a few recently installed ring grinders, Rather moderate grinding conditions are used in the pocket grinders, absorbing not over 150 hp. per pocket with 25-in. wood. Artificial stones of "N" grade and 7 structure are used. Our recent preference has been for aluminum oxide abrasive with an average grit size of 85. Burring is with an 18-cut spiral burr with $1^1/_2$-in. lead.

Screening is in three stages with 0.050-in. perforations in both primary and secondary screens. Flat screens are provided for rescreening of secondary rejects. Extensive use is made of centrifugal cleaners.

The freeness test is the principal control test. An hourly test is run on a composite sample for control purposes. Whenever the freeness falls outside a prescribed range of about 8 points, change is made in either grinder pressure or in stone surface. Pit freenesses are run once per shift and are of use in determining which stone needs adjustment when the regulator freeness shows a change to be necessary.

Handsheet tests are made on regulated stock once per shift. Attention is given to burst, bulk, and fiber fractionation but none of these are regarded as control tests. Increasing attention is being paid to the air porosity test but we have not as yet attempted to use it as a control test.

All of our groundwood is bleached. In the past, emphasis has been placed on obtaining high unbleached brightness in order to reduce bleaching costs. As we have speeded up our paper machines, runnability of the groundwood has become increasingly important and we now realize that certain past practices aimed at reducing decay and discoloration in storage such as the use of sap peeled wood are detrimental to good runnability. We are now endeavouring to provide a uniformly high moisture wood for grinding with the realization problems in wood procurement may result.

Our groundwood has the following approximate test values: TAPPI burst factor 17; TAPPI tear factor, 32; breaking length, me. 3900; bulk, 2.20; freeness (C.S.), 70; per cent passing 150-mesh screen, 45; porosity (Sheffield), 140; unbleached, brightness and 62; bleached brightness, 69.

C. A. Richardson (Blandin Paper Co.) (*Groundwood for Supercalendered Magazine Paper*): Supercalendered publication grades are characterized by several properties which may be grouped as optical, printing, strength, and runnability. In the optical group, color, brightness, opacity, and cleanliness are important. Color and brightness are closely associated. Brightness generally runs in the vicinity of 70, and is readily obtained by bleaching the groundwood pulp together with the use of today's high brightness chemical pulps and filler clays. Color is generally a matter of preference by the customer, and usually runs on the blue-white

side, obtained by the use of blue and purple dyes. Opacity is important, since these grades are generally light in weight, ranging from 35 to 40 lb. basis weight. Opacity is maintained by keeping the groundwood content high, the choice of chemical pulps of relatively high opacity, the use of relatively high clay contents, and by avoiding crushing the sheet on the supercalender. Cleanliness is maintained by the use of fully bleached chemical pulps, and by producing clean groundwood. The printing properties are smoothness or finish, gloss, and ink receptivity. These are important, and are obtained by the production of suitable groundwood, the maintenance of good formation on the paper machine, and careful supercalendering. The proper moisture content and the use of steam showers on the calender are important in obtaining high smoothness and gloss without blackening. Runnability on the printing presses is very important in this grade of paper since it is printed on high-speed presses, either letterpress or rotogravure. Moderate bursting and tensile strength are required, but the most important single strength test is the dry tear test. Burst and tensile come primarily from the groundwood, with tear derived chiefly from the chemical pulps, although the tear strength of the groundwood contributes.

Groundwood pulp is a high percentage of the fiber furnish of these grades. Therefore its properties are important factors in the final sheet.

Cleanliness starts at the woodroom. The wood is thoroughly cleaned of all bark, and rigid inspection insures clean wood to the grinders. Efficient screening removes further dirt. Bleaching further reduces the dirt count. Many mills are going to centrifugal cleaning to further contribute to clean pulp. The presence of shives or slivers in the groundwood is very undesirable. Shive elimination starts at the grinder, through the choice of stone grits and burrs, and the proper dressing of the stones. Proper screening and the use of centrifugal cleaning further remove shives. Thorough refining of screen rejects is important to avoid returning shives to the system after they have once been removed.

Brightness is obtained by peroxide bleaching. In order to maintain a paper brightness off the paper machine of 73, a bleached groundwood brightness of about 69 is required. This is obtained by maintaining an unbleached brightness of about 60 to 61 by proper supervision of the proportions of woods of different ages delivered by the wood yard, and then bleaching in a medium density bleach plant, using about 1.5% Na_2O_2 (applied as a mixture of sodium and hydrogen peroxides).

Good wood, as freshly cut as possible, is required for good groundwood. Spruce is the chief species, making up about 65% of the total wood, the remainder being about evenly divided between balsam and aspen. Each species is ground separately, with stones and grinding conditions chosen for that species. The different pulps are then mixed in the bull ditch.

Four-pocket grinders, each powered by its own synchronous motor are used. The motors vary in size from 650 to 1000 hp., and have a speed of 240 r.p.m. The stones are 28 in. wide to grind 25-in. wood, and are 60 in. when new.

Four types of artificial stones are used, with different grit distributions and compositions for different purposes.

The coarsest stones (Norton 37C544-07V), with 25% each of 46, 60, 70, and 100-mesh grit and an 0 hardness are used with a 10-spiral, $3^1/_2$-in. lead burr for spruce. The pulp is long fibered and strong, and the power consumption is low. These stones make good pulp also at higher freenesses, such as are required when heavier paper is being made on the paper machines and higher freeness and production are required.

Another crystalon stone (Norton 37C605-07V), has 25% each of 60, 80, 90, and 100-mesh grit and 0 hardness. This type of stone is used with a 10-spiral, $3^1/_2$-in. lead burr for spruce and balsam, and with a 10-thread, $^1/_2$-in. lead burr for aspen. The spruce and balsam pulps are somewhat finer than from the coaser stone, with good strength and a slightly higher power consumption.

A third stone (Norton 37C806-N7V), has 50%, 80-mesh and 25% each of 70 and 90-mesh grit and N hardness. It is used with a 10-thread; $^1/_2$-in. lead burr for aspen. The wood is ground at freenesses of 100 to 110 as against 80 for spruce and balsam, and the power consumption is about 75 hp.-days per ton as against 60 to 65 for spruce and about 68 for balsam. The aspen pulp has moderate strength, and adds to the opacity and bleachability to the mixture.

The fourth type is an alundum stone (Norton A602-N7VG), has 50% 60-mesh and 25% each of 70 and 80-mesh grit, and N hardness. It is used with a 12-spiral, $3^1/_2$-in. lead burr for spruce. The pulp is very well suited for this type of paper, with very low rejects, fine fiber, and good strength. The power consumption is moderate, and the production is good.

The pit density is 3.5%, the grinding pressure 30 or 35 lb. depending on the size of the motor, and the temperature between 145 and 150°F. The spruce and balsam are ground at pit freeness if about 80, and the aspen at 100 to 110. The freeness of the unbleached groundwood after the deckers is 70 to 74.

The pulp is screened on Impco screens with 50-cut plates, and the tailings are rescreened on 50-cut plates, with the final rejects going to a Haug refiner.

The same type of pulp is also suitable for machine finish book paper, and for coating base stock.

W. J. Walker (Champion Paper and Fibre Co.) (*Tailoring Groundwood for Coating Body Stock*): The groundwood produced at The Champion Paper and Fibre Co. mill at Pasadena, Tex., is used primarily on one machine producing body stock for *Life* magazine. The sheet is 40-lb. 25 × 38—500 basis. The raw stock is 27 lb. and 13 lb. of coating is applied on the machine.

The daily groundwood production is about 50 tons per day. It is produced on two 5-ft Roberts grinders, each powered by a 2350-kw., 300-r.p.m. motor. The stone size is 62 in. diam. by 66-in. face.

The groundwood furnish range is 45 to 55%. The current machine speed is 1450 f.p.m. and the trimmed sheet is $174^3/_4$ in. wide.

Quality Requirements

Cleanliness. Requirements: The stock must be as free as possible of coarse fiber bundles which would show through the coating.

Control: This accomplished by using fine screen plates 0.040-in. perforation followed by Centri-Cleaners. The screen consistency must be kept at a high figure, 0.75% moisture-free. The screened stock is tested regularly by sampling on a test flat screen to check on operation and possible screen plate breakage or leakage.

The amount and kind of fiber present in the "A" fraction (20-mesh) of the Bauer-McNett classifier is used as a guide to the operations. This should be 1% or less. It is necessary to watch the wood supply as to age as the deterioration during the summer is very rapid. The "blue glass" is used for a general evaluation of the pulp characteristics.

Freeness. Requirements: It is necessary to control the freeness within close limits especially when 50% groundwood or more is used in the furnish. It should not vary more than 10 points in order to permit satisfactory machine operation at a speed of between 1400 and 1500 f.p.m. The range that has proved most satisfactory is from 75 to 85 ml. Canadian standard. If it is slower than this, it does not blend well with the chemical pulp and will not form properly. If it is too free, it will not felt properly and will not give a slow enough mixture.

Control: Freeness is taken at the stones; after the Centri-Cleaners, after bleaching, and as delivered to the meter of the machine after storage.

Limits are set on stone freeness. These are comparatively

wide. The main control is after the Centri·Cleaners and comparatively narrow limits are used at this point.

The load on the grinders is used as a first control when changes are indicated. The stone surfaces are kept in such shape that raising or lowering the loads on the grinders will take care of the normal variations in wood quality or burring cycles. As only two grinders are involved, the stone surfaces must be kept within very narrow ranges for satisfactory performance.

The "blue glass" is used for a first check on pulp appearance after a stone has been treated. A slide projector and microscopic examination are also used to some extent.

Blending and Felting. Requirements: It is necessary to have the pulp in such a condition that it will blend properly with the chemical pulp which, in this case, is semibleached pine kraft and still control the drainage on the wire.

Control: It is found that the bursting strength indicates this property quite well. If the bursting strength is high enough, satisfactory results are obtained. The control is with the horsepower-days per ton input. This ranges from 100 to 120.

Brightness. Requirements: It is necessary to have a 65 G.E. minimum groundwood brightness in order to give a satisfactory brightness in the finished sheet.

Control: Peroxide bleaching varying from $^1/_2$ to $1^3/_4$%, depending upon the wood quality, satisfies the requirement.

General. It may be of interest to note that the wood now being used is cottonwood. Pinewood was used prior to June, 1956. The results with cottonwood are much superior in all respects to those obtained with pine.

R. W. Leaf (Shawano Paper Mills) (*Groundwood for Towelling and Tissue*): Groundwood pulp intended for use in towelling and tissue papers can be quite a different material than the groundwork pulp we've been talking about so far particularly good pulp that will be eventually used in the printing process. In general, softness, absorbancy and bulk are of primary importance in towelling. The paper must be strong enough to function properly, but that doesn't ask too much from it. In certain applications color is important, while in certain other types of towelling it is of very minor importance. In taliing about towelling today and about groundwood to be used in its manufacture I am talking about towels normally made with groundwood contents in the range of 50 to 80%. This is pretty much the typical manila towel. In the case of towelling, wet strength of the finished paper is more important than its dry strength. The towel must be strong enough to hold together while it is used and while it is being taken from the dispenser. In the operations with which I am familiar the strength of the wet web on the paper machine is of minor importance, and the groundwood need not be particularly strong in order to assure good paper machine operation. A towelling grade groundwood should be very free and should contain a modest amount of fines. This will assure good softness, bulk, and absorbancy in the towel. In my opinion groundwood pulp for towelling should have freeness of at least 180 ml. and actually, I would prefer a pulp substantially more free than that. Although shivs are not desirable, they are not as serious a problem as they would be in printing papers. A rather shivvy pulp can be used in the manufacture of towelling because the crepe and embossing patterns do tend to mask the shivs to a certain extent. Large shivs are more serious when there is a large degree of contrast between the color of the chemical pulp and the groundwood than they are when the colors are very similar. Some towels are made from mixtures of unbleached kraft and groundwood and the shivs are much more important than in an ordinary sulphite groundwood towel. Because high freeness pulp is desirable, it is possible to grind wood such as poplar for this application and to do so in reasonably good rates of production. An excellent grade of groundwood towel can be made from 100% poplar. The resulting pulp can be used to make an ideal sheet of manila towelling stock, with excellent softness, bulk, and absorbancy. The good bleachability of this pulp is important for many of the full bleached grades of towels too, and in many of the toilet tissues. I do not believe that much special attention need be paid to the strength of groundwood pulp for towelling, as the basis weights involved and the percentage of groundwood normally used, there should be plenty of strength for good machine operation. I think that a towel with a good finished web strength can be made economically, using the wet-strength resins for strength and using a groundwood pulp with only very modest strength. The freeness must be maintained at a reasonable level so that you do have uniform drainage on the machine wire. Now, in talking about the tissues, there are two different types: the type where groundwood is used as a filler in quantities of 20% and 30% I will talk about first. In those cases you have a high content of chemical pulps and the strength of the groundwood is not too important, and a pulp very similar to that used for towellings should be suitable except that extremely high freeness would not be desirable. You want a lower freeness in order to have paper formation on the machine wire without having it necessary to hydrate the chemical fibers and thus make a stiff and hard sheet. In those cases where the groundwood content is not over about 30%, I think that a pulp with freeness up to about 180 ml. can be quite satisfactory. In the case of the toilet tissues with high groundwood contents, then the strength of the groundwood becomes very important, both for good machine operation and from the standpoint of the strength of the finished product. In these cases, a groundwood with a freeness and a range of 130 to 150 ml. is frequently used. The use of spruce would be desirable, but in many cases is out of the question from the standpoint of economics because the tissues are in a normally low price range. I do know that there are satisfactory tissues are in a normally low price range. I do know that there are satisfactory tissues apparently being made where the groundwood is 100% poplar.

Chairman: I believe you are producing a groundwood entirely different from what we have been hearing about at these sessions, and particularly from a quality viewpoint.

J. A. McLean (Great Northern Paper Co.) (*Tailoring Groundwood for News*): Recent increases in newsprint machine speeds have raised questions regarding the quality of groundwood necessary for potential operation at 3000 f.p.m.

Somewhat similar questions confronted groundwood personnel of the early twenties when the 1000-ft. speed barrier was approached and crossed. With the old 2-ft. grinders, sectional burrs and natural pulpstones of that day, stock variations were the rule. Quality as such was determined by blue glass or the roar of the mill superintendent as he watched broke pile high in the machine aisles.

Thirty-three years later, the 2000-ft. speed barrier was crossed with much less confusion. Technology had added a magic touch to mill equipment and personnel.

At the present time—during this Third International Pulping Conference—there are a number of newsprint machines operating successfully in the 2200 to 2400 f.p.m. range. To say that improvements in groundwood fiber alone are responsible for these speeds or that further control of groundwood of itself will permit substantially higher speeds, would of course be presumptuous. As long, however as it forms a major part of the news furnish, groundwood fiber must be carefully nurtured.

The tailoring of groundwood pulp for the production of newsprint should be along those lines which will give good running conditions on the machine. Normally this calls for a maximum average length for fibers and fibrils with a minimum of diameter to permit flexibility for greater entanglement on the fourdrinier. A reasonably large number of "fines" are necessary for closing up the sheet and providing a uniform formation.

To obtain fiber length, practical experience dictates that component parts of a groundwood mill, such as grinders, prime

movers, load controllers, pulpstones, bearings, lathes, burr mandrels, screens, thickeners, etc., must be well maintained. Improvements in pulp depend on this maintenance just as surely as one leg helps support a tripod.

The second leg of groundwood quality is provided by the experience, skill, and imagination of the operators. From study and association, they have evaluated such basic fundamentals as grinding consistencies, temperatures, and pulpstone grit combinations. They know only too well that a roomful of the wrong stones can be costly and time consuming before replacements are made. The operators are constantly vigilant in regard to proper stone sharpenings and are ready to take corrective steps should the blue glass reveal a resulting trashy pulp.

By using imagination, the operator can visualize what happens at the actual points of contact between particles of grit and wood in the grinding zone where the high temperatures evolved tend to loosen or plasticize the lignin bond between fibers for good separation. This viewpoint can continually assist him in "balancing" grinder production against pocket pressures and stone impressions to obtain pulp of desired characteristics.

The third leg of groundwood quality for news is testing and control. This starts with measurements of the soundness and moisture of incoming wood and follows through the entire process with frequent determinations and recordings of freeness, fractionation, strength, and consistency.

Further specific qualities for news production depend upon a variety of factors concerning cleanliness, furnish, machine characteristics, wire length, speed, vacuum equipment, pick-up, and press arrangements, a full discussion of which would take considerably longer than the time available.

In closing, I might add from personal experience that a satisfactory groundwood pulp for two machines running side by side at 2200 f.p.m. consists of a freeness between 80 and 95 Canadian Standard with a burst of 10 points, tesile of 3500 yds., and tear of 24 g. An average fractionation retained was 4% on 14-mesh, 19% on each 28 and 48-mesh, 16% on 100-mesh, and 42% through 100-mesh screens. Balance of furnish for these machines consists of 20% or less sulfite and 25% chemigroundwood of 250 to 290 freeness. Further increases in speeds up to 2500 f.p.m. on these machines are anticipated with present ground wood quality and furnish.

Speeds of 3000 f.p.m. for standard news production are definitely in the picture in this industry and groundwood operators should constantly collaborate with machine room and control personnel to achieve this goal.

Mr. Cook (Consolidated Water Power & Paper): I would like to ask Mr. McLean just in a broad, general way the characteristic changes in a groundwood required as you advance your speed from, say, 1500 up to 2200.

Mr. McLean: It was quite surprising. Freeness, of course, is a word that we have heard a lot about in these last few days. We found that if we maintained the freeness at the 85 to 100-ml. range, and tried to get a good, maximum average length of fiber with a minimum of diameter, which on the blue glass shows up as fine floating fibrils without the coarse material that usually sinks to the bottom or accumulates on the shoreline as you tile the glass —then we found we had a flexible type of groundwood that has been quite successful in the 2200 f.p.m. speed range. This is the same type of groundwood which also seemed to perform best on slower machines in the 1000 and 1200 f.p.m. range.

Chairman: If I may interrupt, Jim, I believe that as you increased your machine speeds you also had chemigroundwood in the furnish. I believe your machine speed-up program and your chemigroundwood came about at the same time. Am I correct there?

Mr. McLean: That is correct. When we started we had a few troubles. They were due to several factors. Our machines were new to us and to our crews. Our chemigroundwood was new and a considerable amount of development work was necessary in cooking and processing this fiber in relation to our increases in machine seeds. As we became accustomed to handling it, we found that chemigroundwood was a definite help in the manufacture of high-speed news. The high freeness also of the composite pulp furnish to the stuffbox at the percentage of chemi that I mentioned previously does assist in drainage on the wire at these speeds. And, finally, I believe that the adhesion or loading characteristics of the chemi tend to give us a stronger dry finished sheet of news.

Mr. De Montgomery: I would like to ask Mr. McLean the dates and also the locations of these two famous breakthroughs through the 1000 f.p.m. barrier and the 2000 f.p.m. speed barrier.

Mr. McLean: The first machine breaking the 1000 f.p.m speed barrier, on news is generally accepted to be no. 9 machine at Millinocket which reached that point in April of 1921 and carried on successfully at that range. This was reiterated years later by a machinery builder's advertisement in the *Pulp & Paper Magazine of Canada*. If I may be permitted, I will digress by saying that I have a fond spot for no. 9 machine as it was on that machine back in 1927 that I started my papermaking carreer as a fifth hand. The 2000-ft. speed barrier was crossed in February of 1954 at Powell River on their no. 8 machine, I believe, which operated without a pick-up felt. One of the boys from Powell River is here and in discussing this speed with him last evening he substantiates the advertisement of 4 years ago in the *Paper Trade Journal*.

Mr. Kirkpatrick: Jim, did you find when you raised your speed above 2000 that you had to increase the percentage in chemigoundwood and that sheet in order to hold the same freeness relation?

Mr. McLean: The percentage of chemi has been varied as were the other ingredients. We held pretty close to the 25% mark when we were approaching 2000 and have continued on that same percentage in the 2200 f.p.m. range. We have made changes in the chemi cooking and grinding which, we believe, further assisted us in the closed formation which we have had at East Millinocket over the past 6 to 9 months.

Chairman: I have a question for Jim Parsons. Did you mention earlier that you are tending to go to higher moisture wood to improve your groundwood quality, and I believe you stated that that would increase bleaching costs. Is that correct? I would have thought that it would have been the other way, because the greener the wood, as a rule the greater it is, and when you start with a higher initial brightness you should decrease bleaching costs.

Mr. Parsons: Maybe so. We are quite a long way from wood supply in Central Wisconsin. Of course, if we can get all of the rough poplar used inside of 3 or 4 months from the time of cutting, we will have better brightness. However, if some of the rough poplar should be held as long as we are presently holding peeled poplar, maybe up to a year, we are going to suffer loss in brightness.

Mr. Robertson: I have a question for Mr. Walker. I wonder if you would like to elaborate a little bit on this subject of unsatisfactory blending. I never heard of this as a problem before. Would you mention causes and effects? Is it simply a matter of pulps being so flocculated that they won't disperse and mix together, or what are the characteristics of this condition?

Mr. Walker: The blending refers to the way the groundwood and chemical pulp go together and stay together after the stock leaves the slice and moves down the wire to the suction boxes.

If the groundwood is too slow it does not form properly with the chemical pulp. This condition may be due partially to the need of using chemical pulp which is not prepared primarily for this type of sheet. Modifications are necessary in order to satisfy requirements on the other machines.

Mr. Robertson: It was simply the fact that I had equated in my mind blending with mixing when in effect you mean felting down. It is a different concept.

Mr. Walsh: I believe that Bill means how the sheet is formed on the wire, not the blending, necessarily, of the fibers in the chest—how they matt together on the wire. Am I correct there, Bill?

Mr. Walker: That is right.

Mr. Natwick: I think nearly everyone agrees that for groundwood, it is better to grind a higher moisture wood than a dry wood, and we have been doing a little experimental work this past summer trying to introduce moisture back into wood which had dried out considerably. In other words, we were using peeled aspen at 20% moisture, and we sprayed out piles practically all summer long. We did manage to bring that moisture content back up to as high as, in some cases, perhaps as high as 35% in a somewhat spotty fashion, and I am not sure that our efforts were crowned with any great deal of success as far as the quality of the pulp produced was concerned, and I am just wondering if anyone here has done any of this, attempted to reintroduce moisture into the wood and, if so, do they feel that it was successful in terms of results achieved?

Chairman: First of all, could you tell us what the original moisture was. You brought it up to a 35% level, but what was the original moisture?

Mr. Natwick: We were grinding wood of roughly 20% moisture.

Mr. Walsh: We have had some experience along those lines

lthough we didn't try to increase the wood moisture through any particular means other than the usual drive. We had some particularly dry wood which had been fire killed and it was driven down our wood limits, down the river, on an average of about 40 miles, I would say. It did increase the wood moisture. We know that. Bug in my opinion and, there again, it is just an opinion, it didn't do anything to improve the quality of the groundwood.

Mr. Clark: I think all of us will recognize that once green wood has dried you get an irreversible shrinkage that you can't bring back by soaking in water. In the last few years I have been working with structural board made from wafers which are cross-cut particles about 20 thousands thick maybe 2 in. long and half an inch wide, and I find even with these, which are really extraordinary coarse groundwood, that boards made from green wood, give a very much better strength than if the wood is dried and then remoistened to the same degree. There is no doubt that there is an irreversible effect when wood is dried down.

Another point: I notice with interest that for the high-speed newest machine, Mr. McLean's freeness, is somewhere around the order of about 90. Now, I think if he will remember back to the days in 1925 and 1926 it was a rare thing in this country and in the United States, to have groundwood made with a freeness less than 100 C.S. I think there was a survey made in 1925 and it showed that the groundwood made in all the mills ranged from about 105 or so up to 180 and, in those days, the machines were running well below 1000. Now we are up to 2000 and the freeness is well below 100 and I am convinced that when we go up to 3000 the freeness is not going to go up, it is going to go down if anything. I am happy to hear Mr. McLean talk about fiber diameter and fiber length because they, gentlemen, are the keys, greater length, and greater fineness of the fiber that are the qualities that permit us to go to higher speeds. Here, with a freeness of around 70 C.S. Jim Parsons is making coated paper and I am sure he is not making it at 2200 f.p.m. Here we have a freeness of 90 and with the machine going at 2200 to 2300 f.p.m. If we are going to base our philosophy of how to make better groundwood on this freeness-burst-tear-system we are not going to get very far. Advances will be by accident rather than by design.

Mr. Walsh: Thank you, Mr. Clark, although I would like to point out that there has been a little bit of sulfite put in the sheets since we speeded up from 1000 to 2000 ft! I would like to ask Mr. Brecht—I believe he has done considerable work on trying to remoisten wood—would he care to comment on his experiments?

Prof. Brecht: We have carried out some work in this direction. We cut trees and brought the logs to the laboratory within about 24 hr., and were thus able to carry out grinding on the same wood after various stages of drying. We found essentially that there appears to be a minimum moisture content of the wood which will allow a regain in properties on moistening, and this minimum is in the region of about 16 to 17%. It is necessary of course to carry out the remoistening very slowly so that the wood is able to swell again. If the wood is dried to below this minimum, remoistening does not effect a regain of the properties obtained in groundwood from wood of the same moisture content. If, however, this minimum is not exceeded, then remoistening does enable one to obtain properties similar to those obtained with wood of the same moisture content. These results were very gratifying to us, since normally our wood is rather dry, of the order of 22 to 26% moisture content, and moistening enables us to enhance the properties of our groundwood through the production of a higher proportion of long, fine, flexible fibers.

Mr. Walsh: Could you tell us, when you remoistened the wood what was the final moisture? What moisture did you actually use when grinding the wood? Did you bring it up to your usual standard of 30% moisture?

Prof. Becht: More than that, up to 50 or 60 % on occasion. The natural moisture content of the wood on cutting was about 40%, and we dried it out and then brought it back to 40%, and in some instances still higher, by impregnating under pressure.

Mr. McLean: Do you feel, Mr. Brecht, when you are grinding the remoistened wood that the lack of fiber quality is due to the hardness or lack of resiliency of that remoistened wood as compared with the original elasticity?

Prof. Brecht: I'm afraid I cannot say. We were rather surprised by our results. We had in fact expected to lose the desirable properties irreversibly by drying the wood, and the unexpected results were gratifying from the application standpoint.

Mr. De Montmorency: I have some information about the remoistening of logs which is second-hand, I must admit, which I obtained in Scandinavia last year, where some experiments had been done to try to remoisten actual bolts by letting them lie in ponds and spraying them for quite a considerable length of time. This proved to be of little value. The wood didn't dry out any more but it didn't get much wetter either. However, when the wood was actually sunk in the water and kept completely covered it was possible to raise the moisture content from about 20% to about 40%. Unfortunately, this took about 6 months. Grinder experiments on the remoistened wood, moving up from 20 to 40%, showed that either all or a very great deal of the ability to produce good groundwood was regained. I understand that the moisture content had not gone below about 20%. Just recently, personally, I have had occasion to grind just a little bit of sprucewood at about 30% moisture on the miniature grinder, and got a rather discouraging result, particularly in the tearing resistances of the pulp. I remoistened this wood to 55% by sinking it in water. The tear was not quite doubled, but it came up to over the usual 0.5 figure and all the other properties increased very nicely. It made what seemed to be a rather hopeless case a quite practical one.

Mr. Foote (Consolidated Water Power & Paper): I should like to ask Mr. Brecht whether his remarks on remoistening are applicable to hardwoods just the same as softwoods?

Prof. Brecht: We have no information on this point. Our tests were carried out on spruce only.

Mr. Clark: May I just supplement those remarks of Mr. Brecht? Let us be clear on this point, that if you don't dry the wood down further than within the order of 16 or 17% of moisture, you can remoisten and get back what you lost, but if you go below that point then you can't recover since the process is irreversible. I think that may clarify some of these discrepancies discussed.

Mr. Walsh: There is a question, Mr. Brecht, of the time required to resoak these logs?

Prof. Brecht: We carried out the remoistening in a pressure vessel, and the time involved was about 3 or 4 days.

Floor: How much pressure?

Prof. Brecht: I am sorry that I have not the figures with me to answer that question.

Mr. Walsh: If there are no further questions I believe Mr. Quinn has a few closing remarks.

Mr. Quinn: Thank you very much. I think you have conducted a very interesting session and due to the fact that we have another official function coming up at 6 o'clock I don't think we should waste any more time around here. This culminates a very interesting session and I just want to say a word of thanks to our two speakers, Mr. Hunter and Mr. Hoholike, panel members, and Ed Walsh for the excellent work and imagination and effort that they have put into this session.

Two-Stage Groundwood Bleaching

H. F. JACQUES and R. W. BARTON

The possibilities of combining the peroxide and hydrosulfite processes for bleaching mechanical pulps have received attention in the literature by Richardson (*1*), Armstrong (*2*), Smedberg and Statler (*3*), and Barton (*4*). Basing their deductions on laboratory data, these investigators have concluded the following:

The two bleaching processes can be combined to increase the brightness of mechanical pulps to a higher value than can be obtained with either process alone in a single stage. The procedures reported involve either a peroxide bleach at medium density followed by sulfur dioxide neutralization and then treatment at low density with hydrosulfite, or bleaching with hydrosulfite at low density, washing and then treating with peroxide at medium density. Sulfur dioxide neutralization is used after the first-stage peroxide bleach to remove residual oxidizing agent which would react with the hydrosulfite. When hydrosulfite is applied in the first stage, the stock is washed to remove hydrosulfite decomposition products which would consume the peroxide added in the second stage (*1–4*).

In the data reported, commercial groundwoods made from spruce, aspen, and mixtures of spruce and aspen have been bleached to the 75 to 80 brightness range. Brightness increases of 14 to 22 points were obtained with these samples. Final brightness values of 68 to 78 have been obtained with southern pine groundwood and with a large number of pulps made from water, caustic, or bisulfite pretreated chips of beech, birch, balsam, gum, white oak, Douglas-fir, and white

H. F. Jacques, Finch Pruyn and Co., Inc., Glens Falls, N. Y. and R. W. Barton, Virginia Smelting Co., New York, N. Y.

Fig. 1. Two-stage peroxide hydrosulfite bleaching system

fir. Brightness increases exceeding 30 points have been attained in some cases with mechanical pulps made from chips pretreated with alkali (*3*). In the results reported, hydrosulfites provided from 3 to 10 points of the brightness increase and the remainder was supplied by the peroxide (*1–4*).

The cost of bleaching to high brightness levels with the two-stage process is about $1.00 to $1.10 per point of increase per ton of pulp treated. The cost for increasing the brightness above 11 points is less with the two-stage process than with peroxide alone.

The purpose of this paper is to present the results obtained using the combination process to bleach groundwood pulp on a mill basis. In the particular application which will be described, the combination process was used to meet brightness specifications during a period when the wood being processed resulted in pulp which was difficult to bleach with peroxide. The stock was made from a wood mixture containing 70% spruce and 30% balsam fir.

DESCRIPTION OF BLEACHING OPERATION

A flow diagram of the two-stage peroxide-hydrosulfite process used is shown in Fig. 1. The process consisted of the following operations:

(a) Groundwood pulp from the decker storage chest was metered into the system and dewatered to 12 to 15% on a vacuum thickener.

(b) Stock from the thickener was heated with steam to approximately 90 to 110° F. in a double-shaft mixer and then treated with peroxide bleach liquor in a second mixer. The amount of peroxide added was adjusted as required to give a pulp of about 71 brightness.

(c) The heated and chemically impregnated fiber then dropped into a down-flow retention tower where from 2 to 4 hr. of retention were obtained. The amount of retention time was controlled to give a residual of 10% of the initial peroxide concentration.

(d) The stock was diluted to 4 to 5% consistency with fresh water and with white water from the paper machines in an agitated chest located directly beneath the tower. Sulfur dioxide was injected into the stock in this chest to give a pH level of 6. The sulfur dioxide reduced all the residual peroxide and simultaneously lowered the pH of the pulp, preventing darkening of the stock by residual alkali from the bleach.

(e) The stock was then passed through a consistency regulator and into a bleached-pulp storage chest containing a fan-type agitator which circulated the stock around a mid-feather. A sodium hydrosulfite-sodium tripolyphosphate solution was added through a perforated pipe located across the intake of the circulation agitator.

(f) After a retention period of 15 to 30 min., depending upon bleached pulp demand, the stock was metered into the machine chest. Aeration caused by violent agitation in the machine chest oxidized any hydrosulfite remaining in the pulp, thus preventing further bleaching. The temperature of the pulp was from 90 to 95° F. in the hydrosulfite stage.

Table I.

	Bleaching stage	*G.E. Brightness*	*Brightness increase*
60-Day average values	Unbleached	62.7	0
	2.9% Peroxide	71.0	8.3
	2.9% Peroxide +1.0% hydrosulfite	74.4	11.7
Maximum brightness value (one-day average—six measurements)	Unbleached	63.0	0
	3.1% peroxide	73.5	10.5
	3.1% Peroxide +1.0% hydrosulfite	76.9	13.9

(g) The sodium peroxide solution was prepared in batches using a mixture of sodium and hydrogen peroxides. The chemical concentrations based on the moisture-free pulp were from 2 to 4% sodium peroxide, 4.0% sodium silicate (41° Be.) and 0.08% Epsom salts. From 1.6 to 2.0% alkali as NaOH was used depending upon the peroxide concentration.

(h) The sodium tripolyphosphate-sodium hydrosulfite bleaching solution was prepared continuously with two dry-feeders having dissolving basins connected in series. Sodium tripolyphosphate was dissolved first in the water which was then passed into the hydrosulfite basin. Concentrations of 0.50% sodium tripolyphosphate and 1.0% sodium hydrosulfite, both based on the moisture-free weight of the stock, were used.

The process was operated continuously, unbleached pulp being supplied to the peroxide portion of the system and combination bleached pulp being continuously withdrawn from the hydrosulfite system. Metering devices were used to correlate the peroxide addition with the stock rate. No volume control of pulp flow was used between the two stages.

The peroxide system contained all of the essential features for effective bleaching, i.e., medium consistency, through mixing of the bleaching chemicals and the pulp, adequate retention time and good control of the process. The hydrosulfite portion had provisions for rapid, thorough and free mixing and air-free retention, conditions which have been shown necessary for effective hydrosulfite bleaching by Virginia Smelting Co. (*5*), Back (*6*), and Sparrow (*7*). The retention time and temperature were, however, to low for the most economical operation (*4*).

RESULTS

During the period in which the two-stage bleaching system was operated, the brightness values of the unbleached pulp, the peroxide bleached pulp and of the combination peroxide-hydrosulfite bleached pulp were measured. The data obtained were averaged over 10-day periods to equalize varia-

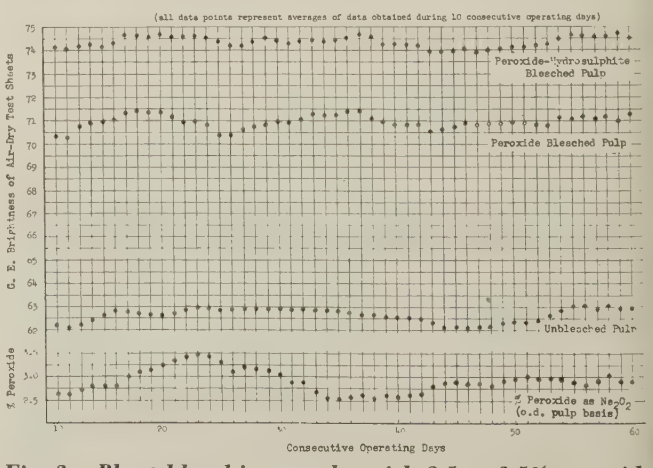
(all data points represent averages of data obtained during 10 consecutive operating days)
Peroxide-Hydrosulphite Bleached Pulp
Peroxide Bleached Pulp
Unbleached Pulp
% Peroxide as Na2O2 (o.d. pulp basis)
G. E. Brightness of Air-Dry Test Sheets
% Peroxide
Consecutive Operating Days

Fig. 2. Plant bleaching results with 2.5 to 3.5% peroxide followed by 1.0% sodium hydrosulfite

tions resulting from deviation in tests procedures and short-term changes in operating conditions. These 10-day average results for a period of 60 days are plotted in Fig. 2. The 10-day averages of the peroxide concentrations used are also plotted on this graph. The numerical averages of the brightness values and peroxide concentrations for the entire 60-day operating period are summarized in Table I. As shown here, 62.7 brightness pulp was bleached to 71.0 with 2.9% peroxide and to 74.4 with 2.9% peroxide plus 1.0% hydrosulfite.

No correlation between the amount of peroxide used and either the first-stage or second-stage brightness is apparent from the plotted data. A reduction in the average peroxide concentration from 3.5 to 2.5% between the thirteenth and twenty-third operating days resulted in a slight increase rather than the expected decrease in the brightness of the bleached pulp. The unbleached stock had an essentially constant brightness value in the range of 62.8 to 63.0 during the same period. These date point out the difficulties in bleaching this particular pulp with peroxide and reflect the extreme changes in bleachability which occurred.

The average brightness improvement with 3.75 to 4.0% sodium peroxide on five isolated days during and just prior to the 60-day operating period was 8.5 points, from 62.1 to 70.6. These results were no better than were obtained with 2.9% sodium peroxide throughout the 60-day period and indicate that, with the pulp used, higher peroxide concentrations would have been of no benefit.

The brightness contribution of 1.0% hydrosulfite combined with 0.50% sodium tripolyphosphate averaged 3.4 points through the entire operating interval. The contribution was fairly constant and variations in the brightness of the pulp after the second stage correlated well with changes in the brightness of the pulp leaving the peroxide tower.

The amount of brightness increase from the second-stage hydrosulfite treatment agrees favorably with that predicted from published laboratory results (*4*). The effect of hydrosulfite reaction time at a temperature of 140°F. is shown in Fig. 3; and the effect of hydrosulfite reaction temperature using 1 hr. time is given in Fig. 4. Both of these curves were plotted from laboratory data obtained using 3.0% peroxide in the first stage. The pulp was made from woods of approximately the same species and geographical origin as those used in the mill.

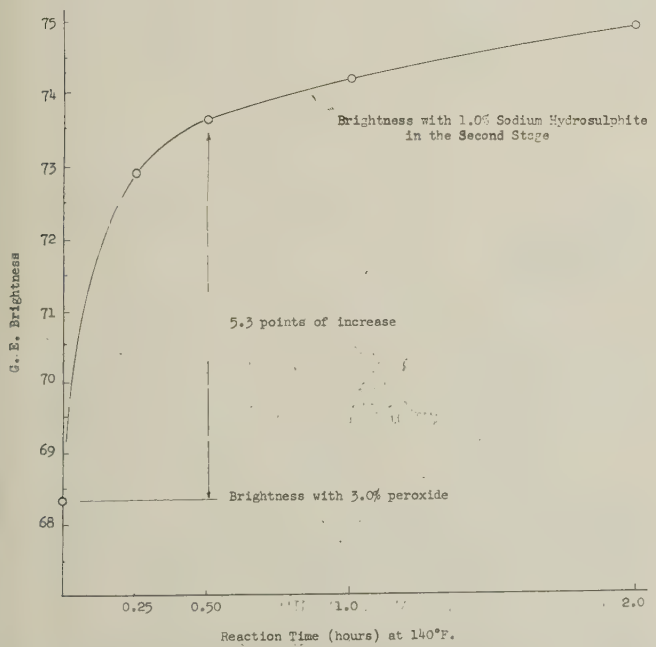

Fig. 3. Effect of hydrosulfite reaction time in two-stage peroxide-hydrosulfite bleaching of groundwood pulp

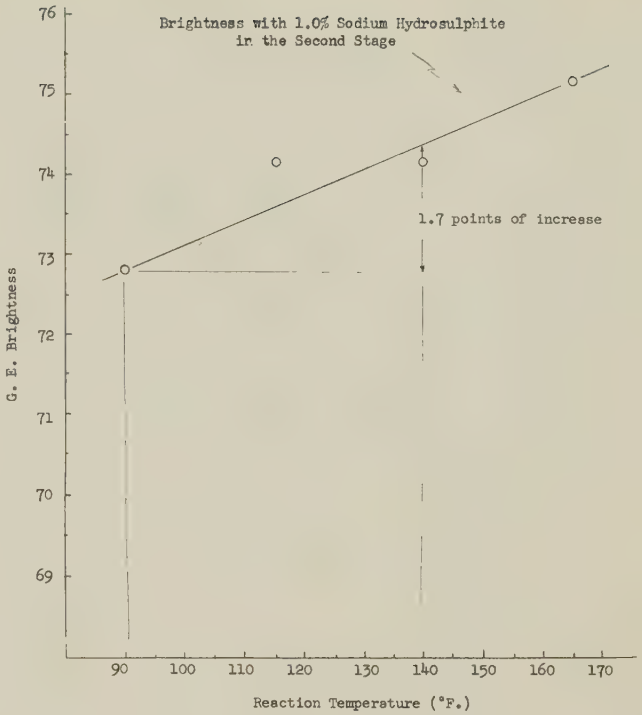

Fig. 4. Effect of Hydrosulfite Reaction Temperature in two-stage peroxide-hydrosulfite bleaching of groundwood pulp

Extrapolating the information presented on these two curves to the conditions used in the mill, i.e., 90°F. and 15 to 30 min. retention time, shows the improvement in the hydrosulfite stage to have been close to the 3.4 points obtained in the mill. In Fig. 3 an increase of 4.7 to 5.3 points in 0.25 to 0.5 hour retention time at 140°F. is indicated. Subtracting 1.7 points, the amount of brightness sacrificed by bleaching at 90 rather than 140°F. for one hour, from this value leaves an expected increase of 3.1 to 3.7 points.

Also included in the summary table are the maximum brightness results obtained during the 60-day operating period. A maximum of 76.9 was attained throughout one day with 3.1% sodium peroxide followed by 1.0% hydrosulfite. The brightness after the peroxide stage was 73.5. The increase from the hydrosulfite stage was the same as obtained throughout the trial, 3.4 points. From these results, it can be concluded that if peroxide bleached pulp values in the range of 73 to 75 had been made, final brightness values from the two-stage system would have been in the range of 77 to 80 or equivalent to those reported in the literature (*1–4*).

COST OF BLEACHING

The costs of chemicals and steam for bleaching with 2.9% peroxide and with 1.0% sodium hydrosulfite are given in Table II. The total cost of 8.3 points of increase with peroxide was $13.44 per ton of pulp; the cost for 3.4 points with hydrosulfite was $5.65; and with the combination, 11.7 points required $19.09. On a per point of brightness-increase per ton of moisture-free pulp bleached basis, these figures are equivalent to $1.62, $1.66, and $1.63 for the peroxide, hydrosulfite, and combination treatments, respectively.

The costs are about 50% higher than the $1.00 to $1.10 reported in the literature (*1, 3, 4*) because of the poor response of the pulp to peroxide and because restricted operating conditions were used in the hydrosulfite stage. If a reaction time longer than 1 hr. and a reaction temperature of 120°F. or higher had been possible, a brightness increase of 5 points would have been obtained in the second stage at a cost approaching $1.10 per ton-point. Had 5 points been obtained,

less peroxide would have been necessary in the first stage. The last or highest points are the most expensive to get with either stage.

Since 11 points of brightness increase could not be achieved

Table II. Cost of Bleaching (Moisture-free pulp basis)

Material	*% Used*	*Pounds per ton pulp*	*Unit Cost[a] (Cents per pound)*	*Chemical cost per ton of pulp*
2.9% Peroxide				
Sodium peroxide	1.3	26.0	0.204	5.30
Hydrogen peroxide, 50%	1.4	28.2	0.211	5 95
Sodium silicate, 41° Bé.	4.0	80	0.016	1.28
Epsom salts	0.08	1.6	0.049	0.08
Sulfur dioxide	0.60	12	0.0463	0.56
Total—Peroxide stage chemicals				13.17
Steam (90 to 110° F. @11% consistency)		364	0.00075	0.27
Total—Peroxide stage				13.44
1.0% Sodium hydrosulfite				
Sodium hydrosulfite	1.0	20	23.75	4.75
Sodium tripolyphosfate	0.50	10	0.04	0.90
Total—Hydrosulfite stage				5.65
Total—2-stage bleach				$19.09

[a] Cost—delivered in car or truckload quantities in Central New York.

under any practical conditions with peroxide alone, no cost comparison between the peroxide and two-stage systems for achieving increases of 11 to 12 points can be made. There is no doubt, however, that the cost would have been substantially less for the two-stage system than for the single-stage treatment had the amount of peroxide necessary to get 11 points been used, i.e., in excess of 4%.

SUMMARY AND CONCLUSIONS

The application of the two-stage peroxide-hydrosufilte process was found to produce brightness results which were approximately 3.5 points higher than those which could be obtained with peroxide alone. This is in agreement with the labor-story data presented in the literature. In addition, a final pulp brightness in the range of 75 to 74 was consistently obtained. The cost of bleaching with the two-stage process was found to be $1.63 per point of increase per ton of moisture-free pulp bleached, which was higher than previously reported but lower than would have been the case if only a singly stage had been used. To have gotten the most economical operation, the hydrosulfite stage should have been operated at temperatures above 120°F. and with a reaction time of 1 hr. or more.

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Reductive Bleaching of Mechanical Pulp with Sodium Borohydride Exploratory

W. C. MAYER and C. P. DONOFRIO

Sodium borohydride, a reducing agent possessing unusual and unique properties, was first synthesized in 1943. It aroused much interest in preparative organic chemistry and is now rapidly attaining important stature in industrial chemistry. Some segments of the pulp and paper industry have already applied the compound to several problems, viz., the alkali stabilization of cellulose by Meller (*10, 11*) and pulp brightness stability by Rapson (*12*). There are perhaps a number of other problems and needs in the industry to which borohydrides can be applied. In this investigation an endeavor was made to explore the bleaching potentialities of sodium borohydride on coniferous mechanical pulp.

COLOR COMPONENTS IN WOOD

Although not fully understood the color of wood or mechanical pulp is linked with certain reactive groups on the benzene ring, in lignin thought to be phenolic or enolic, which have an effect on the resonance of the system (*2, 15*). Brauns (*2*) reported that by covering the phenolic groups in chemical pulp an increase in pulp brightess of about 17 points was obtained. Jones (*7*) relates that there is strong evidence that at least a portion of the phenolic hydroxyls and carbonyl groups in extractable native lignin are largely responsible for the capacity of groundwood to react with peroxide during bleaching. The *alpha* carbonyl group, if present at all in lignin *in situ*, has been regarded as a possible chromophoric structure in treated and extracted lignins; the effects of this group on color properties, however, would be exhibited to greater degree in the ultraviolet range than in the visible region.

Freudenberg (*3*) has postulated quinoid structures arising from an enzymatic action on coniferyl alcohol which in wood, under the proper conditions, will form an amorphous polymerizate called "dehydrogenation polymerizate" (DHP). DHP shows extensive similarities to spruce protolignin and Braun's native lignin. Spectral work carried out on the quinoidal system has revealed high absorption in the 400 mμ range with high shoulders of absorption carrying well into the visible range (*1*). Since most quinoidal structures are easily reduced it would follow in accordance with Freudenberg's hypothesis that pulp reflectance in the visible region would be increased if mechanical pulp is reacted with an effective reductive system.

SODIUM BOROHYDRIDE

Sodium borohydride, $NaBH_4$, is available at a purity of about 98%, as a white crystalline solid. It is stable indefinitely in dry air and decomposes slowly in moist air giving up hydrogen.

The standard e.m.f. of the half reaction (*6*):

$$BH_4^- + 8\,OH^- = BO_2^- + 6\,H_2O + 8e$$

has been calculated to be +1.23 v. versus the standard hydrogen electrode.

The specific reaction of sodium borohydride is the reduction of the carbonyl group of aldehydes, ketones, and acid halides:

$$4\,R_2CO + BH_4^- + 2\,H_2O \rightarrow 4\,R_2CHOH + BO_2^-$$

Carbonyl groups of acids, esters, and acid anhydrides are not attacked by sodium borohydride. Jensen (*6*) points out that certain peroxides are reduced by borohydride.

Numerous papers have been published on the reduction of

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sugars, quinones, carbohydrate degradation products in wood, and other compounds by borohydride in both aqueous and non-aqueous media (*6*).

In aqueous solvents (water, water-alcohol) above pH 9 the borohydrides are stable. As pH is lowered hydrolysis takes place according to the over-all reaction:

$$BH_4^- + H_3O^+ + H_2O = 4\,H_2 + B(OH)_3$$

Jensen (*6*) who made a comprehensive study of the stability of buffered and alkaline solutions of borohydride concluded that besides being first order in both borohydride concentration and in hydrogen ion concentration, the hydrolysis is temperature dependent. When dissolved in pure water the hydrolysis of borohydride causes a rise in pH which is attributed to the formation of basic metaborate ion and consequently, the stability increases.

It has been found that the hydrolysis of borohydride, is also catalyzed by certain metal salts especially those of iron, cobalt, copper and nickel (*14*).

MATERIALS AND PROCEDURES

Materials

The mechanical pulp used in this investigation was obtained from the Sealright-Oswego Falls Corp., Fulton, N. Y. It consisted of 65% red spruce, *Picea rubens*, Sarg., and 35% balsam fir *Abies balsamea* L. When in wet laps of about 55% moisture content its brightness was determined to be 53%.

The bleach chemical sodium borohydride, 98% pure, was supplied by Metal Hydrides Inc., Beverly, Mass.

The compounds below were used in the proportions shown for establishing the desired buffered pH levels in bleaching:

pH 11—7.2 g. of disodium phosphate to 0.8 g. of sodium hydroxide
pH 9.2—sodium tetraborate
pH 8.7—2.5 g. of boric acid to 8.0 g. of sodium tetraborate

Procedures

Bleaching. Pulp bleaching was carried out at 5.0% consistency, unless specified otherwise, in a temperature-controlled water bath capable of maintaining the prescribed temperature within ±1.0°C. In all cases 50 g. of pulp on the moisture-free basis were placed in a 2-l. vessel and all chemicals except sodium borohydride were added. After thorough mixing and attainment of the prescribed temperature the desired weighed-out quantity of dry borohydride was applied to the slurry and vigorous mixing was applied for a span of exactly 1 min., the end of which was considered zero bleaching time. In bleaches where buffers were used, the following amounts were added per liter of pulp slurry:

pH 11.0 buffer—6.5 g.
pH 9.2 buffer—8.0 g.
pH 8.7 buffer—20.0 g.

Sampling. In order to facilitate the evaluation of both the rate of borohydride disappearance and corresponding pulp brightness changes throughout the course of bleaching it became necessary because of lack of time between sampling intervals to save pulp samples with drawn for hand sheet preparation until the end of the bleach. Where this was found necessary the bleaching reaction in the sample was terminated by quickly dispersing it, after partial dewatering, into 400 ml. of alum solution at pH 4.5 to 5.0.

Handsheet Preparation. All pulp specimens subjected to brightness evaluation were prepared into suspensions adjusted to a pH between 4.5 and 5.0 using alum. The volume of suspension required for handsheet formation was poured into a clean sheet mold and drained after stirring.

Pulp Brightness. Brightness was measured on both sides of the handsheets after calibrating the Hunter Multipurpose Reflectometer with a 51.3% brightness standard.

Analytical Methods

Gas Evolution Method. One mole of pure borohydride decomposes upon acidification to produce four moles of hydrogen. The simple method of borohydride analysis detailed by Jensen (*6*) is based upon this decomposition reaction.

Iodate Method. The easier and faster basic iodate method described by Lyttle *et al.* (*9*) was revised only to the extent of increasing the concentration of the standard iodate solution to 0.35 *N*. This revision was found to provide the desired ac-

Table I. Sodium Borohydride Consumption and Brightness Increase as Affected by Bleaching pH—1 and 2% $NaBH_4$ Added—35°C.—5% Pulp Consistency

Bleach No.	*$NaBH_4$ added, %*	*Bleaching time, min.*	*$NaBH_4$ consumed of total, %*	*Brightness, %*	*pH*
		Buffered at pH 8.7			
1	2.00	0	0	54.8	
		2	17.4	61.2	
		10	99.4	70.5	
		Buffered at pH 9.2			
2	2.00	0	0	54.8	
		2	26.8	58.1	
		8	47.3	60.1	
		20	79.5	61.0	
		30	90.9	61.9	
		48	99.5	61.8	
		NaOH added to pH 10.0			
3	1.00	0	0	54.7	
		3	5.8	56.4	
		14	25.5	58.3	
		30	40.0	59.3	
		60	56.2	61.2	
		120	79.5	62.1	
		Buffered at pH 11.0			
4	1.00	0	0	54.4	
		2	0	56.8	
		15	16.3	58.3	
		41	25.9	59.2	
		66	30.0	59.7	
		120	38.4	60.9	
		180	53.0	61.4	
		210	57.8	62.1	
5	0.50	0	0	54.9	
		3	14.0	55.2	
		19	17.3	56.7	
		35	23.9	57.4	
		60	29.2	57.4	
		120	40.8	59.1	
		180	51.5	59.3	
		Unbuffered			
6	2.00	0	0	54.8	
		2	24.0	58.8	9.35
		10	31.7	61.2	
		20	49.5	62.0	
		33	59.6	62.6	9.55
		51	72.6	63.6	
		75	82.7	65.0	9.65
		100	87.0	65.4	
		129	93.4	65.2	9.65
7	1.00	0	0	54.8	
		2	30.3	58.1	
		10	39.2	60.1	9.40
		25	65.3	61.3	
		52	83.6	62.4	9.50
		80	95.3	62.7	
		120	98.4	62.4	9.50
8	0.50	0	0	55.0	
		2	29.7	57.9	9.20
		14	75.4	59.6	
		25	83.0	60.9	9.30
		40	91.0	60.9	
		60	97.0	61.2	9.40
9	0.25	0	0	54.5	
		3	44.3	57.7	9.00
		12	77.3	58.3	
		25	91.6	59.0	9.20
		60	98.6	58.8	9.20

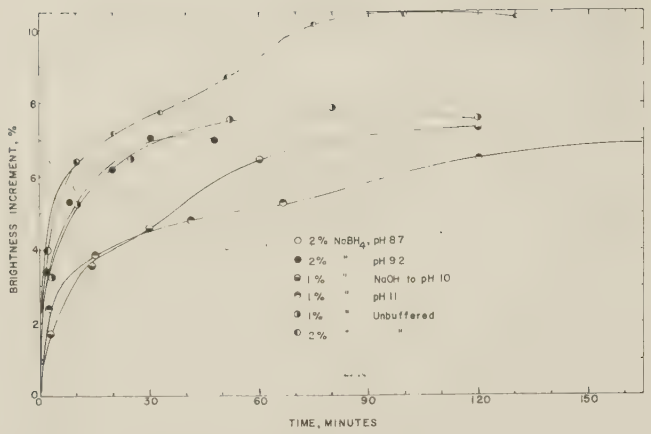

Fig. 1. The effect of pH on brightness development—1% and 2% $NaBH_4$—35°C.

curacy in results when analyzing the high dilutions of borohydride which resulted in the bleach experiments. Both methods were employed in this investigation.

EXPERIMENTAL

Bleaching experiments were designed for investigating primarily the effects of pH, temperature, per cent application of borohydride, and pulp consistency on extent and rates of the reactions, borohydride disappearance, and pulp brightness development. In most instances the sodium borohydride consumption data represent total consumption with no distinction being made between that consumed by the pulp and that lost as hydrogen through hydrolytic decomposition. In a single experiment, however, to be described later, this distinction was made.

Effect of pH

At buffered pH levels of 8.7, 9.2, and 11.0 1 and 2% additions of sodium borohydride, on the moisture-free pulp basis, were made to bleaches conducted at 35°C. and 5% consistency, Table I, Fig. 1. For comparison the data of two unbuffered bleaches and one in which sodium hydroxide was added to provide for initial pH of 10.0 are shown.

In accordance with the foregoing discussion on borohydride stability it can be observed that pH's greater than the unbuffered slow down markedly both reactions, brightness development and borohydride disappearance, and that the over-all brightness increments are not materially improved over those secured with the unbuffered systems; moreover, there is the added disadvantage of time requirements for bleaching. In bleaching at buffered pH levels maintained below those obtainable with unbuffered systems reaction rates are accelerated but without advantages in brightness production or effective borohydride utilization. The maximum brightness increment of 10.5 points was realized through the application of 2.0% $NaBH_4$ in an unbuffered system wherein it was observed that the pH ascended from an initial value of slightly greater than 9 to 9.65. Also, the 1.0% borohydride addition in an unbuffered system (av. pH of about 9.4) provided a brightness gain superior to that secured with 2.0% borohydride buffered at pH 9.2.

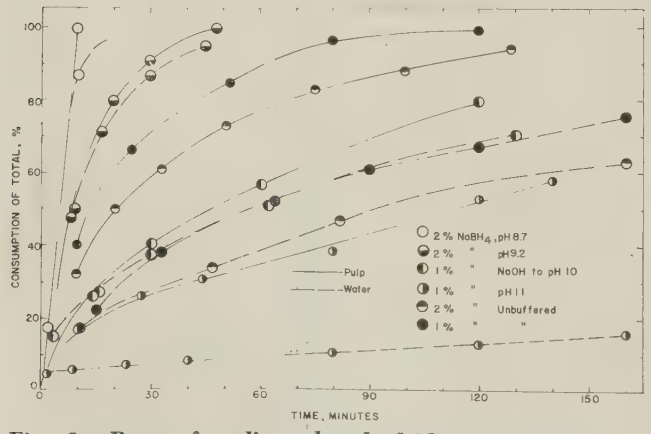

Fig. 2. Rate of sodium borohydride consumption in bleaching and in pulp-free solutions—35°C.

Table II. Sodium Borohydride Consumption in Water of Comparable Concentration and pH as Pulp Bleaches at 35°C., 5% Pulp Consistency

Bleach No.	*$NaBH_4$ added, %*	*Time, min.*	*$NaBH_4$ consumed of total, %*
	Buffered at pH 8.7		
1	2.00	0	0
		10	86.8
		25	99.8
	Buffered at pH 9.2		
2	2.00	0	0
		1	9.0
		8½	48.0
		16	71.3
		30	86.3
		45	95
	NaOH to pH 10		
3	1.00	0	0
		3	15.1
		15	26.7
		30	37.2
		62	51.6
		130	70.4
		182	78.8
	Buffered at pH 11		
4	1.00	0	0
		2	4.5
		13	5.3
		35	7.0
		60	7.8
		120	10.3
		180	12.6
		240	15.7
5	0.50	0	0
		3	2.1
		15	2.9
		31	4.5
		61	6.2
		165	9.5
		240	11.5
		27 Hours	51.7
	Unbuffered		
6	2.00	0	0
		3	13.5
		10	16.0
		47	33.4
		82	46.4
		180	66.4
7	1.00	0	0
		15	21.2
		33	37.5
		64	52.0
		90	60.6
		120	67.0
		160	75.0
8	0.50	0	0
		2	25.6
		11	48.7
		20	54.0
		40	69.3
		60	78.6
		95	87.3
		125	91.8
		150	96.0
9	0.25	0	0
		2	26.0
		15	54.0
		30	73.6
		60	87.7
		120	98.5

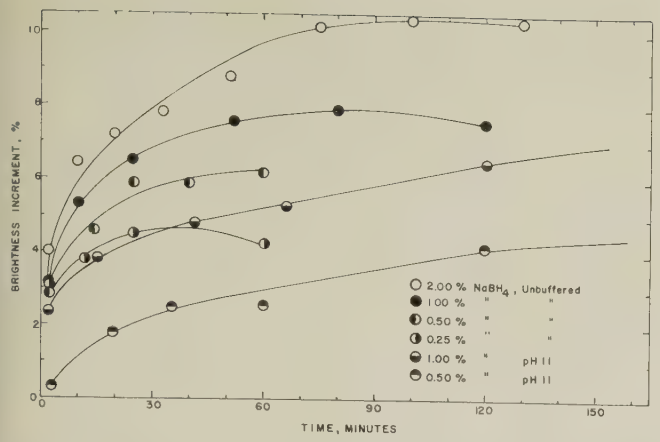

Fig. 3. Relationships of brightness development with different per cent applications of sodium borohydride

The borohydride disappearance data for the above bleaches are plotted in Fig. 2 and are represented by solid lines. Parallel data were obtained for pulp-free systems and these are represented by dotted lines (refer to Table II). It is of interest to note that at pH levels lower than those developed by unbuffered systems the rate of borohydride disappearance is slightly lower for the pulp-free system. Since sizable but not optimum brightness gains were obtained under these same conditions the results appear to suggest that little borohydride is required for bleaching, provided it can be reacted effectively. Only at the buffered pH of 11 was the stability of borohydride observed to be significantly improved over that of the unbuffered systems.

Effect of Per Cent Sodium Borohydride Application

Additions of 2.0, 1.0, 0.5, and 0.25% $NaBH_4$ were made in four bleaches conducted under unbuffered conditions at 35°C. and 5.0% consistency. Two more bleaches under the same conditions of temperature and consistency were carried out in systems buffered at pH 11 with applications of 1.0 and 0.5% $NaBH_4$, Table I. The brightness advancement during bleaching illustrated in Fig. 3 reveals superior performance for unbuffered over buffered bleaches. While the highest brightness increment was indicated for the 2.0% borohydride application it is evident that sizable brightness increments approximating in magnitude those usually observed for commercial mechanical pulps could be obtained with 0.5 and 1.0% borohydride. At the buffered pH of 11 the over-all brightness increment of the 1.0% borohydride bleach is seen to approach that of the 1.0% unbuffered bleach with about two thirds as much chemical being consumed. However, applications of borohydride greater than 1.0% at very high pH's require excessive bleaching time.

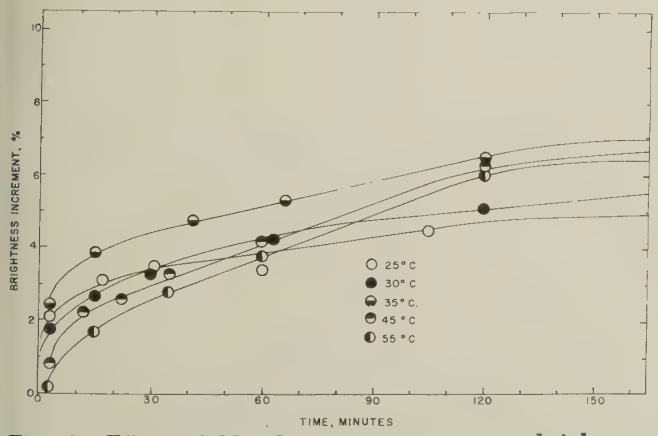

Fig. 4. Effect of bleaching temperature on brightness development—1% $NaBH_4$—pH 11

Effect of Temperature

The effect of temperature was studied over the range of 25 to 55°C. with bleaching systems buffered at pH 11 and employing 1.0% borohydride on the moisture-free pulp basis, Table III, Fig. 4. The buffered conditions were chosen in order to avoid the side effects arising from the normal pH changes which occur in unbuffered systems. Further, the earlier work demonstrated that buffering at pH 11 provided satisfactory chemical stability.

The data on temperature effects were not extensive enough for permitting kinetic analysis. Only the important factors of brightness development and borohydride consumption were observed. Chemical consumption was accelerated by increasing the temperature of bleaching but at the same time adverse effects were wrought on pulp brightness. Optimum performance was observed at 35°C. and the greatest divergency in performance became apparent for the initial phases of reaction where, as the tabulated data indicate for the common point of 20% borohydride exhaustion, the brightness increments for 35, 45, and 55°C. were about 4.0, 2.5, and 0.5 points, respectively. Beyond that point throughout most of the course of bleaching the slope of brightness against chemical consumption remained almost the same. A satisfactory

Table III. Effect of Temperature on Rate of Sodium Borohydride Consumption and Brightness Increment of Pulp—Buffered at pH 11, 1% $NaBH_4$

Bleach no.	*Time, min.*	*$NaBH_4$ consumption of total, %*	*Brightness*
	25°C.		
5	0	0.0	54.0
	3	9.4	56.2
	17	13.4	57.1
	31	16.4	57.5
	60	22.4	57.4
	105	25.8	58.5
	180	32.0	59.0
	240	35.4	59.5
	300	38.4	59.6
	30°C.		
6	0	0.0	53.5
	3	3.8	55.3
	15	14.5	56.2
	30	19.5	56.8
	63	22.6	57.8
	120	31.4	58.6
	180	37.9	59.2
	35°C.		
4	0	0.0	54.4
	2	0.0	56.8
	15	16.3	58.3
	41	25.9	59.2
	66	30.0	59.7
	120	38.4	60.9
	180	53.0	61.4
	210	57.8	62.1
	45°C.		
7	0	0.0	54.6
	3	10.3	55.4
	12	15.7	56.9
	22	19.8	57.2
	35	25.3	57.9
	60	34.8	58.8
	120	51.7	60.9
	180	66.2	61.3
	55°C.		
8	0	0.0	54.9
	3	19.4	55.1
	15	21.5	56.6
	35	30.6	67.7
	60	38.6	58.7
	120	61.4	61.0
	205	80.5	61.4

Table IV. Effect of Pulp Consistency on Rate of Sodium Borohydride Consumption and Brightness Increment of the Pulp—35°C., 1% $NaBH_4$

Bleach no.	*Time, min.*	*$NaBH_4$ consumption of total, %*	*Brightness*
	2.5% O.D. Pulp Consistency		
10	0	0.0	53.6
	5	35.7	58.3
	17	62.6	60.1
	31	81.8	60.7
	61	93.6	61.4
	120	99.6	61.4
	5.0% O.D. Pulp Consistency		
11	0	0.0	54.8
	2	30.3	58.1
	10	39.2	60.1
	25	65.3	61.3
	52	83.6	62.4
	80	95.3	62.7
	120	98.4	62.4
	7.5% O.D. Pulp Consistency		
12	0	0.0	53.9
	5	37.6	58.6
	18	63.4	59.4
	42	82.0	60.7
	63	88.4	61.3
	80	91.7	62.0
	112	97.8	61.8
	10.0% O.D. Pulp Consistency		
13	0	0.0	53.8
	5	33.1	58.3
	15	51.0	60.0
	30	57.5	61.4
	60	80.0	61.4
	125	96.5	61.5

explanation for the unexpected initial lower rates of brightness increase for the higher temperatures employed cannot be offered at this time. Part of the answer may lie in the decomposition histories which revealed pronouncedly greater rates at the temperature levels higher than 35°C. However, interfering reactions occurring in the pulp itself must also be taken into account. It is well known that groundwood in an alkaline medium will darken to a greater extent at elevated temperatures, a color change which some investigators associate with condensation or structural rearrangement of constituents in the pulp system. Additional work would be required to further the knowledge on this important point. It should be borne in mind, however, that the temperature effects reported herein are restricted to a fixed bleaching procedure in which the last step administered was the addition of sodium borohydride. It is probable that different effects may be produced by a different order of addition, specifically, by adding the pulp lastly to the borohydride system buffered at high pH.

Table V. Measurement of Hydrogen Evolution over Sodium Borohydride Bleaches

$NaBH_4$ distribution	*Conditions of bleach: 2.5% O.D. pulp consistency, unbuffered, 35°C., 1% $NaBH_4$*	*5.0% O.D. pulp consistency, buffered at pH 11, 35°C., 1% $NaBH_4$*	*5.0% O.D. pulp consistency, unbuffered, 35°C., 1% $NaBH_4$*
Per cent of $NaBH_4$ measured as H_2 evolved	74.5	31.7	76.7
Per cent of $NaBH_4$ residual	0.0	45.0	5.2
Maximum per cent of $NaBH_4$ consumed by pulp at time of test	25.5	23.3	18.1

Table VI. Actual Rate of Sodium Borohydride Consumption by Pulp as Compared to Brightness Increase. Bleach Made at 2.5% O.D. Pulp Consistency, 35°C., Unbuffered, and 1% $NaBH_4$

Time	*$NaBH_4$ measured as H_2 evolved, % of total*	*$NaBH_4$ consumed in bleach, % of total*	*Maximum $NaBH_4$ consumed by pulp % of total*	*Brightness increment*
0	0.0	0.0	0.0	0.0
5	22.0	35.7	13.7	4.7
17	46.0	62.6	16.6	6.5
31	59.0	81.8	22.8	7.1
61	70.1	93.6	23.5	7.8
120	74.5	99.6	25.1	7.8

Effect of Consistency

The altering of pulp bleaching consistency from 2.5 to 10.0% for the 1.0% application of sodium borohydride in unbuffered systems at 35°C. revealed, as expected, slightly improved chemical stability, Table IV. However, the rate of brightness increase and over-all brightness increments were found not to be significantly different within the range investigated.

Measurement of Hydrogen Evolution During Bleaching

As pointed out earlier one of the products of borohydride decomposition in aqueous solution is hydrogen. However, no hydrogen is evolved from the reduction of carbonyl groups. Further, in all bleaches discussed heretofore total borohydride consumption values were reported. In order to separate the total consumption into its component parts, namely, that evolved as hydrogen and that consumed by the pulp three bleaches were conducted under conditions which permitted the collection and measurement of the evolved hydrogen gas during the course of bleaching. The corresponding borohydride residuals in the bleach system were determined also. Thus, the difference obtained by subtracting from the total borohydride applied the sum of evolved hydrogen plus borohydride residual was designated as the quantity consumed by the pulp. (All chemicals expressed on common basis, Tables V and VI.) Figure 5 shows the consumption histories for both the pulp and pulp-free systems when 1.0% borohydride was applied at 35°C. under unbuffered conditions. Of outstanding interest is the observation that the pulp consumed only one quarter of the borohydride applied to produce about an 8 point brightness increase.

Table V, comparing hydrogen losses for the buffered and unbuffered systems, indicates that for bleaching at a pH of 11 the loss of borohydride is substantially lower than that of the unbuffered system but greater than that measured for a pulp-free system at the same pH.

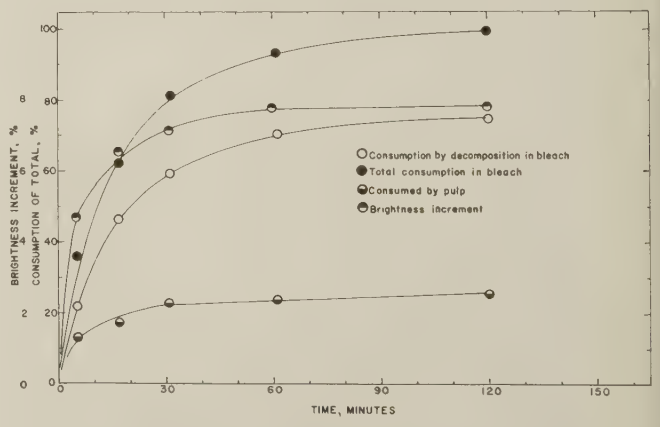

Fig. 5. Sodium borohydride consumption histories—consumed by pulp and by hydrolysis—35°C., unbuffered, 1% $NaBH_4$

Comparison of Groundwood Bleaching Chemicals

The mechanical pulp selected for this investigation was also subjected to bleaching with zinc hydrosulphite and sodium peroxide with the purpose of comparing brightness development and spectral changes in bleached pulps. A 5.0% slurry of pulp was treated at 35°C. with 2.0% zinc hydrosulphite and another slurry of the same solids content was treated at 45°C. with the system consisting of 5.0% sodium silicate (water glass), 0.25% magnesium sulphate, and 2.0% sodium peroxide, all based on the moisture-free pulp. Sulphuric acid was used for adjusting the pH of the peroxide bleach to 10.7. After complete exhaustion of active chemical, pulp samples from each bleach were suspended in water, adjusted to pH 5.0 with alum, and handsheets were prepared. The brightness results shown in Table VII along with those of two borohydride bleaches conducted in the earlier work indicated that the magnitude of brightness obtained with 2.0% $NaBH_4$ is equal to that obtained with 2.0% sodium peroxide. The brightness value shown for the zinc hydrosulphite bleach must not be regarded as being representative as other experimental work in these laboratories has shown that the brightness increment for 2.0% application of zinc hydrosulphite on the same type of pulp is in the order of at least 8 points.

It was felt, however, that the differences in magnitude of brightness would not interfere with a qualitative comparison of the spectral changes conferred on the pulps by the three bleaching agents under investigation, Table VIII. The spectral curves of the unbleached and bleached pulps shown in Fig. 6 were produced from the data obtained with a Beckman model DU spectrophotometer provided with an attachment for measuring diffuse reflectance using a tungsten light source. Calibration was accomplished with a freshly prepared magnesium carbonate surface the brightness of which was taken as 98%.

The data indicate that sodium borohydride produces a distinct advantage in reflectance over the other bleaching chemicals in the 300 to about 425 mμ range and that its bleaching action compares favorably with that of peroxide over the remainder of the spectrum examined (note that 1.0% borohydride bleach is being compared with 2.0% peroxide bleach). Sodium borohydride achieves the reduction of certain groups on which zinc hydrosulphite, also a reducing agent, has only a slight effect as the spectral data in the 300 to 425 mμ range

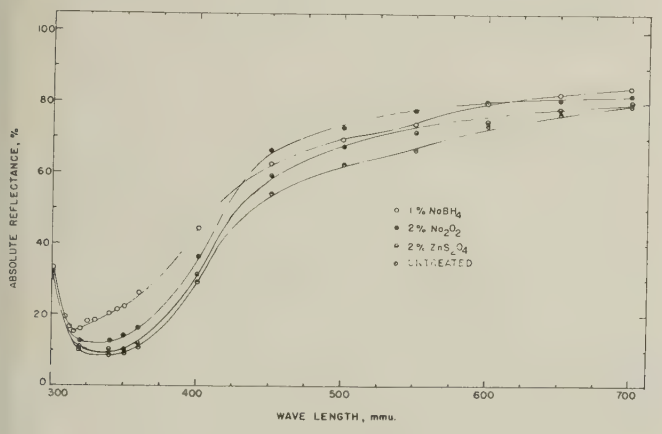

Fig. 6. Absolute reflectance spectra of bleached handsheets

bears out. A borohydride reduction of *alpha* carbonyl groups on the phenyl ring would explain this effect, but as it has been emphasized earlier, the presence of *alpha* carbonyl goups in the pulp under investigation is problematical. Moreover, if the Freudenberg's theory (*3*) regarding the presence of the quinoid structures is considered, it would possibly provide an explanation for spectral changes not only in the lower range discussed but for almost all of the 300 to 600 mμ range.

Ultraviolet Spectrum of Untreated and Borohydride Treated Solutions of Indulin A

As an additional point of interest a lignin preparation, Indulin A, a kraft pine lignin known to contain *alpha* carbonyl

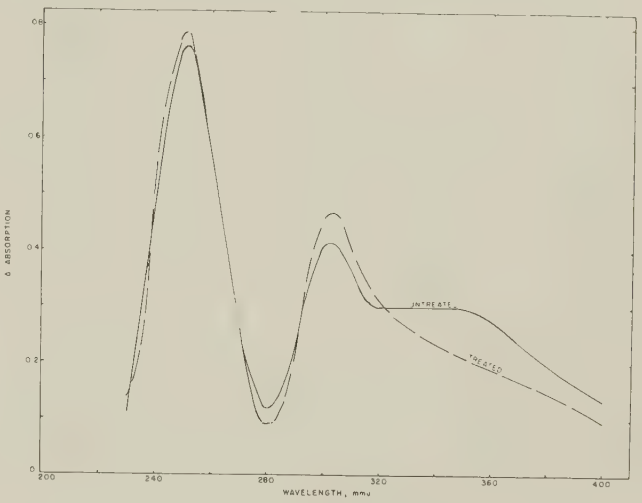

Fig. 7. Difference spectra of sodium borohydride treated and untreated lignin

groups was spectrophotometrically investigated for the purpose of showing the ultraviolet spectral changes wrought by sodium borohydride reduction. Difference spectra curves for untreated and borohydride treated lignin obtained by techniques developed from the basic work of Lemon (*8*) and Goldschmid (*4, 5*) indicated peaks at 250 and 303 mμ for both systems, Fig. 7. The untreated lignin curve exhibits a distinct shoulder or "masked maximum" at about 350 mμ, which, it has been found, is produced by carbonyl groups conjugated with a phenol ring (*13*). However, a peak in the 300 mμ range was reported to be associated with unconjugated phenols. Thus the depression of the shoulder in the 350 mμ range must be caused by the reduction of carbonyl groups. The reduction of *alpha* carbonyl groups should form a greater number of unconjugated phenols and thereby intensify the peak at 300 mμ. There is little doubt that this is the exact nature of the change shown by the curves of Fig. 7 which, further, may be taken as proof that sodium borohydride will reduce at least a portion of the conjugated carbonyl groups present in this form of lignin structure. It is of interest to note, that the spectral changes in the 300 to 400 mμ range correspond to those observed over the same range when the spectral properties of unbleached and borohydride bleached pulps were compared in the previous section. However, it is to be re-emphasized that presence of *alpha* carbonyl groups in lignin, *in situ*, is doubtful.

SUMMARY

It has been demonstrated that sodium borohydride confers a distinct bleaching effect on coniferous mechanical pulp to the extent which compares favorably with that obtained with an equal amount of sodium peroxide. Spectral analysis of

Table VII. Brightness Increments Experienced with ZnS_2O_4, Na_2O_2 and $NaBH_4$ Bleaches

Bleaching agent	*Amount used, %*	*Brightness increment*
Sodium peroxide	2	10.4
Zinc hydrosulphite	2	4.9
Sodium borohydride	1	7.9
Sodium borohydride	2	10.4

Table VIII. Absolute Reflectances of Bleached Handsheets

Wave length, mμ	Reflectance, %			
	Unbleached	2% Na_2O_2	2% ZnS_2O_4	1% $NaBH_4$
300	30.9	32.7	31.4	33.3
310	...	...	...	19.6
312.5	...	...	...	16.2
315	...	...	...	15.7
320	11.3	13.0	11.9	16.7
325	...	...	...	18.1
330	...	...	...	18.6
340	9.3	13.7	10.9	20.6
345	...	...	...	21.6
350	9.8	14.4	10.9	22.6
360	11.3	16.6	12.3	26.4
400	29.4	36.2	32.0	45.0
450	54.8	65.6	59.8	63.7
500	62.7	73.5	67.6	69.5
550	66.3	78.0	72.7	73.8
600	73.7	81.6	75.0	81.2
650	77.9	81.8	79.0	82.8
700	80.4	82.5	80.4	84.2

bleached pulps revealed that sodium borohydride improved reflectance over a wider range than either peroxide or zinc hydrosulphite. The same data indicated the action of borohydride to be distinctly different from that of zinc hydrosulphite, also a reducing agent. Only a small proportion of the applied borohydride is consumed by the pulp during bleaching the remainder is evolved as hydrogen through decomposition. For example, it was shown that when 1.0% sodium borohydride was applied on the moisture-free pulp basis, about 0.25% was consumed by the pulp to produce a brightness increment of about 8 points. Thus, the most important considerations in the utilization of borohydride for reductive bleaching involve the choice of conditions which maintain the greatest stability of chemical and at the same time allow the reductive reaction to proceed at a practical rate. A lowering of pH results in more rapid rates of both the reductive and decomposition reactions. A very high pH is found to improve stability markedly but excessively long bleaching times are required for the attainment of satisfactory brightness increments. Hence, unbuffered systems or systems in which the pH is established at a level near 10 may be found more satisfactory. The maintenance of pH below the level normally resulting from unbuffered and very dilute borohydride solutions as employed in this work are unsatisfactory for mechanical pulp bleaching.

The effectiveness with which borohydride is utilized in bleaching may be greatly impaired by the presence of impurities which catalyzed the decomposition reaction. Thus stabilization of the bleaching system against the action of impurities, a measure found necessary in other forms of mechanical bleaching, may well be a profitable direction for future work.

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Drying Groundwood at Sheet Harbour

R. M. STERNBERGER

During the spring of 1957, we began investigating the various possibilities of drying groundwood pulp with a view to reducing handling and freight charges. At that time we dried mechanically to 56% air-dry on three 120-in. Kamyr machines. The pulp was taken in sheets from the Kamyrs, baled in 500-lb. bales, conveyed by belt some 150 ft. from the mill proper to a weigh shed, thence by fork-lift to storage. We looked at several drying systems then existing and proposed and settled finally on the Bauer-bale system. After discussing our dryer setup, I will go over the reasons for our choice.

THE BAUER-BALE SYSTEM

In this method of drying, the pulp is dewatered mechanically to 40 to 50% solids; "fluffed," dewatered further with heat to approximately 85% solids, and then baled in the fluffed form. The bale, then, is a solid brick or block of pulp, not a pile of sheets. It can best be likened to a bale of cotton.

Mechanical Dewatering

We retained the Kamyr machines to accomplish the mechanical dewatering action. We added cutters and slitters and the pulp, in small sheets of about 8 by 10 in., discharges into a horizontal screw conveyor that runs along the face of the three machines. Besides moving the pulp, the screw conveyor has the additional function of breaking up the sheets and initiating the "fluffing" action. A cleated belt conveyor then carries the pulp up a 30° incline to the fluffer.

Fluffer

The fluffer is a small, nonprecision, single disk refiner equipped with devil-tooth plates. It is fed by a screw-feeder discharging directly into the eye of the disk. The teeth of the plates intermesh and fiberize the pulp by impact. No refining action takes place. The fluffer also acts as an effective air-lock to the drier.

Drier

The drier is a direct-fired rotating drum of the kind used for agricultural purposes for many years. By direct fired, we mean of course, that the drum is heated by the furnace gases themselves and that there is no exchange of heat.

The drum is composed of three telescoping cylinders with compound showering flights. This gives expanding cross-sectional area and provides different velocities and different heats in each of the three cylinders to suit the changing rates of evaporation as the moisture content of the pulp drops.

The pulp falls from the fluffer into the stream of hot gases at the juncture between the furnace and the drum. At the far end, a fan unit pulls the air through the furnace and the pulp and heated gases through the drum. The only other air supply is a small furnace blower for oil atomization.

The fan then blows the pulp and gases to a large cyclone where the pulp is separated from the furnace gases and moist air. This cyclone discharges into a hopper where the pulp is picked up by a second fan and conveyed through a blow-pipe to the baler, a distance of 120 ft.

Control of the drier is very simple. A drier outlet temperature is selected, and the furnace adjusts automatically to achieve this temperature. The drier inlet temperature will vary with the water load to the drier, but the outlet temperature and the moisture content of the pulp remain constant.

We operate at an outlet temperature of 200°F. This gives us an air-dry test of 92%. We can easily dry the pulp further, even to 100%, but we find we get better performance through-

R. M. Sternberger, Vice-President, Halifax Power & Pulp Co., Ltd.

out the system at this moisture content. Under normal operating conditions the inlet temperature is 700°F.

An empirical heat balance indicates a vacuum of about 4 in. within the drier. Apparently then, three factors are helping to dry the pulp: mechanical action (tumbling), heat, and a vacuum.

Baler

The two-story double box baler operates with three 14 in. single-acting rams. As the name implies, there are two bins with floating bottoms that rotate on a center post. While one bin is located under the cyclone collector to receive the pulp being blown from the mill, the other is positioned over the rams.

A tramper packs the pulp into the bin that is filling. Bale weight is controlled by the amount of work the tramper does. In other words, as the bin fills, the tramper must work harder to complete its stroke. Control is achieved by having the tramper stop when it is drawing a certain current. When this occurs, a signal sounds. The two bins are then indexed 180°. This places the full bin over the rams and empty one under the collector. The pulp is then compressed from the bottom and a bale formed. The bale is extracted by opening doors at the top of the bin and the baler is then ready to repeat its cycle. Meanwhile, of course, the empty bin is filling.

We operate in this manner within a 5-min. cycle, getting a 740 lb. bale. This weight gives us three bales to the air-dry ton.

Oil System

Our fuel is Bunker "C," we have found it satisfactory in every respect. Because of our location, it is necessary to take tanker load delivery. To this end, we installed a 24,000 bbl. storage tank, approximately 1200 ft. from our wharf. This is supplied by a 10-in. line heated electrically. The balance of the oil heating system is done by steam. We also installed a 2000-gal. insulated day tank. The oil is preheated to 235°F. before combustion. Consumption averages 15 gal. per air-dry ton.

Installation

On Oct. 11, 1957, we shut down for this installation.

By removing the old balers, room was made available in the existing building for the drier. The fluffer is located directly over the drier. The baler was placed in a small new building, erected at the site of the old weigh-shed. Installation was substantially completed on Dec. 20, 1957.

STARTUP

Because we were pioneering a development we expected many startup difficulties. We were not disappointed.

Dusting

Dusting, or loss of fines, was immediately apparent. Some occurred at the two cyclone collectors and was overcome simply by balancing out the cyclones by adjusting the caps.

The worst situation, however, was at the baler. The action of the tramper was like an air pump, blowing fines through every possible opening. This has been overcome by sealing the tramper section as much as possible; changing the tramper program (slowing it down, etc.) and the installing a simple scavenger system.

This scavenger is merely a vacuum cleaner installed at strategic points around the baler. The fines and spilt pulp that it picks up are blown back to the mill, collected in a small cyclone, and introduced into the white-water system.

Oil Contamination

At startup, particles of unburnt oil were contaminating our pulp. This was not evident to the naked eye in anyway, and none of our standard test showed it up.

This contamination was discovered by one of our customers who, after making a manila liner board using our pulp in the liner, ran the standard solvent test for asphalt in the backing. The solvent caused the oil particles to dissolve and bleed through the liner, giving the board a bad case of measles.

Two simple changes in the furnace overcame this difficulty. We changed the nozzle to give better atomization and we added one course of fire brick at the throat of the furnace to restrict the flow of air.

Bale Density

At the top of the ram stroke, the newly formed bale has a density of 57 lb. per cu. ft. The bale, however, has considerable "springback" and we had trouble finding a method of tying off the bale with a material strong enough to contain this density.

Compounding the trouble is the fact that whatever method is used, it must fit into the time-cycle of the baler. We have settled on a heavy strapping accomplished with pneumatic tools.

Bale Mechanical Strength

While the bale as a whole has good mechanical properties, unprotected corners tend to crumble off with heavy handling. As all of our shipments are by water, it became necessary for us to wrap the bales.

We accomplish this wrapping in the baler with overlapping top and bottom sheets leaving the ends open. We use standard unbleached sulphite.

Everyone concerned is happy about this development. It has eliminated a housekeeping problem for ourselves, our transportation, and our customers.

Reliability

Because the system had never been asked to perform on a 24-hr. basis, many components were not up to the task. This was especially true in the baler.

The weak points have been found out and, of course, corrected.

DECISION FACTORS

Besides cost, several factors persuaded us that this system was particularly suited to our needs.

We felt that the refiner was the best approach to the "fluffing" problem. Other methods have been known to make fish-eyes, but we have never encountered this problem.

At first glance, the direct-fired drier seems impossible but there are four factors in its favor:

(1) Fuel economy. Obviously better than in a system using a heat exchanger.

(2) No warm up necessary. Light the flame and you're ready to go.

(3) Because the pulp is carried through the drier in the furnace gases, and because most of the oxygen has been consumed from these gases, the pulp is dried in a relatively inert atmosphere. In other words, there is little oxygen available to oxidize the pulp.

(4) With proper combustion, there is no danger of contaminating the pulp.

The baler chosen is giving us the size bale, bale weight, and bale density we felt most suited for our needs.

CONCLUSION

Besides the obvious advantage of a freight saving, this drier has permitted us to ship to more distant markets and to ship to and store in warm climates.

Besides the advantages of reduced handling which redounds to the benefit of both ourselves and our customers, our customers enjoy a real benefit in the friability of this pulp. It seems to disperse more readily than wet laps.

In spite of slightly increased manufacturing costs, we are enjoying real economies with this system.

Glomera Pulp Drying System at John Breakey Limited

L. GAGNON AND KARL STANGL

During 1955, the management of John Breakey Ltd. was faced with a difficult decision. Plans were being made to increase the mill capacity from 40 to 80 air-dry tons per day, with the object of shipping all the output in dry form to increase the shipping range and reduce transportation costs. The difficulty was that conventional methods for drying pulp were prohibitively high in capital investment and operating cost, and also met with customer objections. In the search for a more efficient, acceptable, and above all, cheaper method of drying wood pulp, John Breakey Ltd. joined with Canadian Sumner Iron Works Ltd. in investigating the possibility of flash drying the pulp with a Glomera type drier.

PILOT PLANT EXPERIMENTATION

Late in 1956 a complete pilot plant, consisting of pulp fluffer, Glomera drying tower, 25-gal. oil burner, furnace box and Glomera briquettor, was installed at Breakeyville, and after several weeks of experimenting it was established that a flash drier would do a perfect job and cost far less in installation and operation than a conventional drier. It also turned out that the briquettor was not suitable to handle the fluffer pulp. After several unsuccessful attempts an adequate and successful pulp compressor was designed.

Starting up the Plant

The complete drying plant, including the Breakey-Sumner pulp compressor, was delivered and installed at Breakeyville by the middle of August 1957. After several test runs it became obvious that certain modifications were required, especially on the furnace and the pulp compressor. The furnace had to be modified to meet the varying atmospheric conditions and has lately been replaced. The pulp compressor had to be arranged to take care of the extremes in mill output dictated by mill or market conditions. The modifications to the furnace and pulp compressor were completed late in October at which time the first car of flash dried, fluff groundwood pulp was shipped.

L. Gagnon, Mill Superintendent, John Breakey Ltd., and K. Stangl, Vice-President, D and S Engineering Ltd.

THE GLOMERA PULP DRIER

The drying is performed in three stages. The equipment employed consists of an oil-fired furnace, a Novorotor double disk pulp fluffer receiving pulp at 30% air-dry consistency. Through the fluffer the pulp is brought in contact with hot gases and in conjunction with the discharge fan, the fluffer acts as first drying stage. A 10-ft. cyclone separates the evaporated water from the pulp and completes the first drying stage. The pulp then enters the second stream of hot gases and is carried through a type 1500 Glomera flash drier, separated from the steam and gases in a 12-ft cyclone, and introduced into the third stage drier which is a duplicate of the second. The drying completed, the pulp enters the compressor through a plexiglass chute which gives the operator and opportunity to observe the quantity and condition of the pulp entering the compressor and make the necesary adjustments to the system.

Hot Air Furnace

Hot gases were originally supplied by a pair of Iron Fireman rotary cup Bunker "C" oil burners with a capacity of 100 U. S. gal. per hr. for each burner. They are now produced by a specially designed Raskin hor air double shell furnace fitted with a 200 U. S. gal. per hr. Raskin mechanical atomizer burner. Diluting air is mixed with the combustion gases at the outlet of the furnace to reduce the temperature to a point suitable for drying. This diluent air is preheated by passing between the shells of the furnace. Fuel used is Bunker "C" oil delivered to the mill daily by truck.

The Drying System

The drying unit itself is a suspension type flash drier. This means that the material to be dried is carried through the system by the drying medium, either combustion gases in an open system, or hot air in a so-called closed system. As soon as the material comes in contact with the gas, most of the heat exchange takes place and the surface water is flashed off. The drying tower presents the necessary time element to permit a complete heat exchange, which is necessary to extract some of the fiber bound moisture. During the entire process the material is not in lasting contact with the mold structure of the drying equipment, avoiding any charring and roasting effects which would be damaging to the pulp. There are several factors affecting the drier. The most important are: the initial moisture content of the pulp, the atmospheric condi-

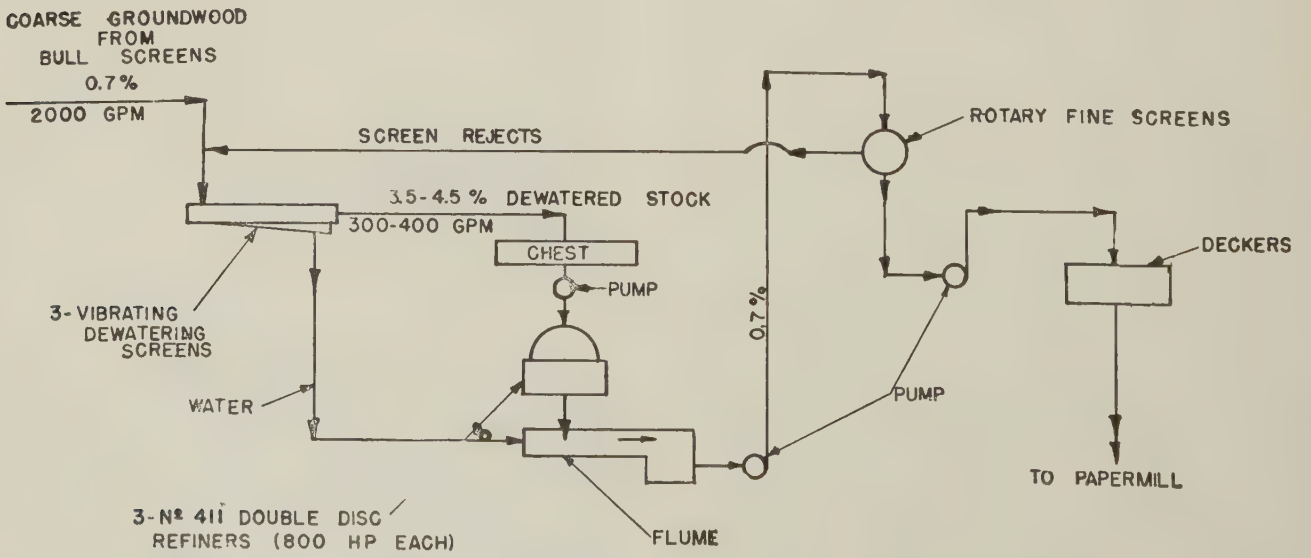

Fig. 1

tions and the quantity of pulp processed. All are automatically controlled through Bailey equipment by varying the burner output and the amount of diluent air used. The dry material is separated from the steam and gas mixture by a series of especially designed high efficiency cyclones.

Pulp Compressor and Baler

The pulp compressor built by Canadian Sumner Iron Works Ltd. is a piston type compressor fitted with an extra large fly wheel, adjustable compression box and d. c. variable-speed drive. It has a speed range of 14 to 45 strokes per min. which represents minimum and maximum capacities of 30 and 90 air-dry tons per day.

The dry pulp is discharged in sheets of 24 by 12 by 1 in. The sheets are made into bales on a modified wet pulp hydraulic baler. The bales are 24 by 36 by 40 in. and weigh 550 lb. They are strapped with standard $^3/_4$ by 0.015 in. steel bands and shipped in box cars. A standard car of 25 air-dry tons contains 104 bales.

PULP CHARACTERISTICS

All the characteristics of the pulp are retained in this drying process. The fluff-dried pulp is remarkable for the ease with which it can be repulped and the resistance to molding and freezing. In sheet form, it handles relatively well and is most convenient in preparing paper machine furnish.

OPERATING DATA

The John Breakey Ltd., Glomera pulp drying unit is the first of its kind. It has been in operation less than a year during which time it has undergone several modifications. It is therefore impossible to offer any definite and completely reliable data. The following represents extremes met during the few months of operation.

	Minimum	*Maximum*
Capacity per day, air-dry tons	35	125
Inlet stage temperatures	400°F.	550°F.
Air-pulp mixture at inlet to cyclones	110°F.	160°F.
B.T.U. per lb. of water evaporated	1200	1600
Consistency of dried pulp	70% a.d.	94 % a.d.

EXPANSION TO EXISTING GROUNDWOOD MILL

At the same time the drier plant was installed, the output of the mill was doubled by the addition of four electrically driven pocket grinders, a Cowan screen, a new bank of Bauer cleaners and modifications to the white water, the stock and the refining systems. Wet machines were converted to deckers to remove water from the screened stock prior to feeding it to a Kamyr vacuum decker. This vacuum decker delivers pulp at an average consistency of 18% a.d., and is followed by two P and L disk type rotary presses, which bring the pulp to about 30% a.d. ton for conveying to the drier plant. This mill expansion together with the installation of the drier plant and auxiliary equipment was completed without the loss of production.

NEW COMPRESSOR AND BALER

D and S Engineering Ltd. of Vancouver, B. C., who now handle all Glomera and related equipment, have designed and built a new completely hydraulic pulp compressor and baler producing 35 by 24 by 1-in. sheets which are automatically made into bales 18 in. high. This machine is now under test in Norway. It has a wider capacity range than the Breakey-Sumner pulp compressor.

The authors wish to thank the management of John Breakey Ltd., Canadian Sumner Iron Works Ltd., and D and S Engineering Ltd. for their very generous cooperation in the designing and starting up of this new type pulp drier and also the Executive Committee of the Third International Mechanical Pulping Conference for the opportunity to present this communication.

MR. O'NEILL: May we have some questions from the floor? Mr. Clark and Mr. Sternberger, here are two questions. First of all, I think you said that you used $17^1/_2$ gal. of bunker C oil to air-dry a ton. Do you happen to have the B.t.u. per pound of water evaporated as compared with the other system, handy?

MR. STERNBERGER: We are not completely certain of the B.t.u. content of our oil but I do believe that we average about 1500 B.t.u.'s per pound water evaporated.

MR. CLARK: Now, the other question is, have either of you gentlemen made any measurements on the loss of strength of the pulp—let's say either burst or tensile?

MR. STERNBERGER: I am not convinced that there *is* any loss of strength. Certainly characteristics of the pulp are changed by drying but that shouldn't surprise us. I don't think that it is an irreversible process, and I believe the pulp can be returned to its original state by hydration. I like to think that the change is a matter of fiber shrinkage.

MR. CLARK: Well, you don't have any data then, to indicate, for example, the effect on density, the test sheet density, or specific volume?

MR. GAGNON: No. I must say that on market groundwood pulp we mostly depend on customers' acceptance and we have had no reports of any drop in srength from customers.

MR. CLARK: Well at least I should think you have some very nice customers. There is probably at least a small drop, but the interesting part about it is, you know, you may be able to dry groundwood without a marked loss of strength but not with the chemical pulp, at least, with the beaten chemical pulp so well, because then you will really reduce your strength.

FLOOR: I notice you mentioned you didn't think there was any change in the properties through this flash drying.

MR. OVERSTROM (Gulf Pulp): Well, I was very disappointed. Last summer we sent some pulp to Springfield, Ohio, to be flash dried and when it was returned the strength had dropped considerably, 20 to 30%, and the freeness had gone up about 50%, so I presume there is some losses of fines causing that. And, also, you were very kind, both of you, to send me some pulp samples of the pulp before and after this flash drying and I must say that the tests fully confirmed our previous results. I sent some of these samples over to England to our customers and they wrote back and said "We certainly don't like to have that kind of pulp." So, I don't know.

MR. STERNBERGER: I want to try to clear up one thing. We labored under the misconception for quite a time that this change in the pulp, where the most noticeably apparent thing is increase in freeness through drying, was due to the loss of fines, but we have satisfied ourselves now that it is not due to the loss of fines. We think it is a fiber shrinkage and it is not an irreversible thing. As I say, you can return the fiber to its original state by soaking it.

MR. CROSBY (Dorr Oliver): I might make a comment or two here. I think Dr. Clark's point is very well taken. It certainly depends on the type of fiber with which you are working. At one time I did considerable work on very careful air drying, this time, of chemical pulps, and we found that you reached a point in the drying state where the fiber temperature started to go above the normal wet-bulb temperature. This was in the range of 78 to 85%, moisture-free fiber. Beyond that point we got what we considered to be an irreversible loss in strength. Prior to that point we got no measurable loss in strength. I might say that I am not intimating at all that this would occur or be true with groundwood, but that was the case on sulphite pulps. Not a great loss, but it was there and measurable.

MR. LOGAN (Anglo Paper): On the question of increased freeness and loss of strength, we have done a very, very cursory test on the pulp which we were fortunate enough to get from Breakeyville and, for the informaion of the audience, generally, it is well to note that if you test this pulp immediately mixed up in water, you do get a very, very marked increase in freeness and you get a very, very marked decrease in strength properties. However, if you mix it up in the Waring Blendor for a relatively short time, a very high percentage of this change is recovered. I do not have the figures at my fingertips, nor do I believe that the samples that we got are necessarily absolutely matched, however, there was not such a very large difference in the final results. Possibly, Fritz Luhde might recall the figures, or have an idea of them.

F. LUDHE (Anglo Paper Products, Ltd.): Two samples were to be tested. The wet sample had been taken just before the stock enters the drying system; the dried sample had been taken at the outlet of the drying system.

A previous experiment showed that the wet sample, after a short soaking time and disintegration, had a freeness of 250 ml. C.S.F. After having been treated for 5 min. in the Waring Blendor, freeness decreased to about 130 ml. C.F.s.

Therefore, I considered it to be a better method to soak both the wet and dried sample overnight. Only a mild and short treatment in the disintegrator was necessary to dissolve the wet sample properly. The dried sample, however, had to be taken into the Waring Blendor as the knots had not been dissolved

during the above treatment. Handsheets, made from this stock, showed numerous knots, uniformly distributed over the whole area. I treated the dried sample for 5 min. in the Waring Blendor until all knots had disappeared. Thereafter, the freeness was lower than that of the wet sample which had been soaked overnight. Tensile and burst were about 10% higher than those of the wet sample. Tear was about the same.

MR. CLARK: May I make one comment on that. Five minutes in a Waring Blendor is a pretty tough treatment. I doubt very much if anyone using groundwood is going to give anywhere near that amount of beating or refining in the mill. I don't care what happens to the freeness. That may go up or down, but the point is that having reached even a higher strength than the original pulp indicated, you are doing a pretty good job of beating in the Waring Blendor. Somebody in the audience may have tried the British standard desintegration treatment, the British standard and have those figures available. I think they will show some decrease.

MR. STERNBERGER: I would like to comment on that. The proof of the pudding is always in the eating, and we have some very nice customers, too, I guess. I think that you could get the same type of results with just soaking. I'll admit that is very strenuous treatment, 5 min. in a Waring Blendor, but all you are doing is accelerating the wetting process. One of our customer's stock spends 5 hr. in his system, from the hydropulper to his cylinder machine, so we soaked our pulp for 4 hr. and we found that just that 4 hr. soaking with no mechanical working returned it to very much its original state, very similar to its original state.

MR. NATWICK: I have a question but I don't know if I ought to break in at this time because it hasn't got anything to do with this subject. If this subject is pretty well exhausted maybe I can come in now. I wanted to ask Mr. Sternberger if you have ever taken your pulp at 40 to 45% consistency, I believe it was, and baled it at that stage, rather than drying it.

MR. STERNBERGER: No, we have not tried that.

MR. NATWICK: I wonder if anyone here has had any experience of that nature, of taking 40 to 45% consistency of pulp and baling it?

FLOOR: You mean baling it in the manner similar to (interruption)?

MR. NATWICK: Fluffed and baled baling it at that moisture instead of carrying it out to the dry pulp? If anybody could answer that later on I would appreciate it. I would like to know what kind of experience they have had with it. I would like to ask Mr. Sternberger or Mr. Gagnon about what it costs per bale to strap.

MR. STERNBERGER: We are not completely satisfied with our present method. It costs too much. At the moment we are using galvanized strap and we are going to use a painted strap eventually, I am sure. I can't tell you per bale but it costs us over a dollar a ton to strap.

MR. NATWICK: Well, that is quite expensive. I want to ask Mr. Gagnon one question. I was wondering, are you satisfied with your press? The reason I ask, I understand from your paper that you are drying your pulp starting at about 30% air dried and I have seen Jackson & Church presses working on groundwood, and that pulp was in the neighborhood of 40% moisture-free. It would seem to me that you have quite an extra expense there, extra water to remove.

MR. GAGNON: Yes, I quite agree with you. It is an additional expense, but on the other hand the Jackson & Church press has the disadvantage of creating nodules in the pulp which are very hard to break. We feel that it is cheaper to dry the pulp from 30% up rather than to have the experience of not being able to break up the pulp after it is dry. The second thing is that our press, as it is installed, doesn't give its maximum efficiency. It is a new press in the paper industry and the company who manufactured it is presently experimenting on various types of feed, and so on. They expect to be able to achieve at least 35%. I think Mr. Dunbar, if he is in the room, would probably have more information in that respect as he has been connected with the tests.

MR. NATWICK: I would like to say this, with regard to the presence of nodules in the pulp with the Jackson & Church press, I realize that they have been a problem. However, I have seen this press working at the Charmin Paper Co. on groundwood and was assured that nodules or "fisheyes" were not present.

MR. GAGNON: Excuse me a moment. I'd say on the wet bulk you wouldn't have too much trouble but once you have dried the nodules they are pretty hard to beat up again.

MR. DUNBAR (DVS Engineering): On the P & L press we are running tests now and we have reached approximately 45% consistency on the outlet of the press. We haven't completed our tests yet but they will be completed shortly and we'll publish the results.

MR. R. O'DONOGHUE: I want to ask Mr. Gagnon about this press. As I understand it, it makes 1-in. sheets and I presume that is by taking a charge and putting a ram against it and then releasing that, and another charge and you get a whole series of disks, or whatever they are. Now, how do you then compress them into a bale and what is the weight of the bale per cubic foot?

MR. GAGNON: The pulp compressor in appearance is just about the same as a bark press, the Ecusta press, I believe. It is a piston type and each stroke produces a sheet. Now, as those sheets are discharged from the press they are filed into a standard-type baler and made into 550-lb. bales. We presently get about 30 lb. per cu. ft. but we have been experimenting with heavier baling presses, such as used for sulphite and kraft, and we have been able to ahieve between 45 and 50 lb. per cu. ft.

MR. STERNBERG: You have given information on the oil requirements for flash drying, but we have no information on the electrical energy required. There are a number of points, especially in the Glomyra system, where electrical power is connected. Could you give us information about required horsepowers per daily ton, or at least how many connected horsepowers you have on the unit?

MR. STERNBERGER: I have some figures that I think you would find interesting. Our work isn't complete on this. We have only had this system for a short time and we have been so busy trying to make it work that we still don't know much about it. We do seem to consume about 7 1/2 hp-days per air-dry ton, and that is dewatering the pulp from 1 to 50% moisture-free mechanically, and then further dewatering by heat to 95% air-dry, or 88% moisture-free. About 3 hp-days per ton are expended in the mechanical dewatering action and the balance in moving the pulp through the drier and then baling it.

MR. GAGNON: We have no definite data on the power but we have, altogether, installed about 600 hp, from the deckers up to the baling end. Now we, presently, are using about 450 to 500 hp. Some of the motors were made oversized because the exact requirements couldn't be determined previously. Now, in our case, the power consumption will vary with the output, as depending on the amount of pulp we are pushing through the system, our horsepower figure will vary. With 500 hp, say, being used, and a capacity of 80 tons per day would give you, probably, between 6 and 7 hp. per ton.

MR. OVERSTROM: Coming back to the quality, Mr. Sternberger said that after 4 hr. soaking it will come back to original tests. Well, we soaked for 48 hr. even then it was a very big difference. While it had improved, there was still a very big difference. We also tried to disintegrate this pulp with hot water and that was an improvement, but we didn't reach the test figures for the pulp which it had before it had been flash dried. There is also another point to consider. You are shipping the pulp, I presume direct to cars, as it certainly can't stand outside storage. I tried a few bales, for instance, in June this year when it was very wet weather. In 2 weeks' time all the bands burst and the bale completely disintegrated, which is a great disadvantage to producers as well as customers.

MR. STERNBERGER: No, that is one disadvantage. These bales will not weather well; they cannot be stored unprotected. On the other hand, the bales that we wrapped with a wrap of sulphite seem to stand up all right. I would like to make one comment on this point. You can't run any system for 5 min. and expect it to run properly. It has to be going, and have a constant heat and so forth. So long as you are sending 700 lb. of pulp to be dried you are never going to get satisfactory results. You would be out of business before you are in business, if you know what I mean, on that 700 lb., that little trial run. Our experience was, at least, as disappointing as yours on the pulp we sent away to dry experimentally. When you get a system going on a full-time basis the results are a little better.

MR. GAGNON: Regarding how the bales stand up in the outside weather: in November last yearwe made a bale with just two straps around, 3/4 by 0.15 in. The bale was left in the open. It is still at the mill if anyone wants to see it. It is yellowed on the outside, but outside of that the bale is still standing up fine and the pulp inside is still a fairly light color.

MR. QUINN (Quebec North Shore): Could you tell me if any attempts have been made to flash-dry sulphite and, if so, what effects the system would have?

CHAIRMAN: Does anyone have any information on drying fluff sulphite pulp?

MR. GAGNON: Our pilot plant was moved to Sumner Ironworks plant in Vancouver, and they have been making trial runs of sulphite and kraft. Probably Mr. Dunbar could comment somewhat on those trials.

MR. DUNBAR: We have completed some trials on unbleached kraft, and we noted an increase in strength. I have the figures if anyone is interested in seeing them. Being at a groundwood convention I didn't bring the figures down with me. On bleached sulphite we have run some tests which indicate no change in brightness. However, we haven't completed our analysis regarding the pulp characteristics after drying.

MR. ANKER: We heard yesterday from Prof. Brecht on the

question of moisture in wood, that when the moisture content went below 17 or 18% that was what we might call a point of no return. You get into an irreversible process. This point, also, is the same which I think we call the saturation point of the fiber which, again, means that this 17% is then contained in the fibers. Now, we have heard from the two gentlemen who have read these papers to us, that they operate with an air-dry content of about 94 to 96. Well, that corresponds roughly to abont 82 to 83% moisture-free. In other words, we are exactly on the borderline which Professor Bates mentioned. So if you go any further, then we are definitely over the danger line, and when you look at the process, how this is taking place in your driers, this is not single fibers which are blown through. They consist of many fibers and, also, the conditions are not necessarily equal all over the place. It comes out at an average which is almost on the danger line, and I think it is very reasonable to suggest that some of these fibers have been going beyond the line and some others are below it, and this might also give the explanation why we find, in some cases, that there is a definite drop in strength. At least, I should like to hear what you think of the conditions, that they might possibly not be so uniform as to give all of it exactly the same drying.

Mr. Cirrito (Rice-Barton): In this connection I would like to ask Mr. Crosby of Dorr Oliver, in the tests that he made on sulphite pulp, just how thick his mat was, and what temperatures he used just to prevent deteriorating the pulp in the high dryness range.

Mr. Crosby: We carried out this work in two different fashions. One involved drying hand sheets, of 350 to 400-lb. basis weight for a fairly long time under carefully controlled humidity and temperature conditions. In the other case we actually set up a device and dried the pulp under very carefully controlled conditions by pulling heated air through a formed sheet. The sheet was formed on about a 14-in. sheet mold and then we would clamp that sheet and pull the air through the sheet in order to get intimate contact with the fibers.

Floor: What air temperatures did we use in drying to the final drying range?

Mr. Crosby: Around 180° would be an answer, but we covered the whole range in the course of the studies.

Chairman: Do we have any other questions on flash drying from the floor?

Mr. Clark: I think Cris Anker has put his finger on this spot. I did not know when we referred to the moisture figures, whether they were on an air-dry or moisture-free basis and I quite agree with him and Dr. Brecht. If you don't bring the absolute moisture content down below 17 or 20% or so you are not going to do any harm to mechanical pulp. You are going to do less harm to mechanical pulp than you will do to sulphite pulp. I remember some sulphite pulp that was made at Anacortes in 1929 and over to England. It was flash-dried pulp and that is the worst pulp, I think, we ever had. It was impossible; however, it was completely dried. You can't reduce pulp to a moisture content of less than 20 or so without harming it to some degree. The figure of 85 is mentioned here. I am prepared to accept that. But we can't dry pulp very much more than that and hope to get a real reversible and recovery of strength properties.

Floor: Mr. Crosby, did you notice any degradation in the strength of your chemical pulps when you dried in the lower ranges, or did you find degradation through the whole range in strength and brightness? I should think that in the 180 or so 200° range you should find it difficult to damage this pulp.

Mr. Crosby: You are asking me to go back a little ways in my memory here, but I would say this,that it isn't so much a matter of the actual temperature as it is a matter of the water removal. We talk, about a so-called "zipper" action when you get beyond a certain dryness. Apparently certain chemical bonds are formed even though you dry very mildly. If you go beyond say, 85 to 87% moisture-free (not air-dry) you tend to get, as near as we could measure it, an irreversible action. The damage comes, of course, when the fiber starts to heat up. We ran into the problem of what we called "case hardening" on the outside of the sheet on the drier rolls, where we would get extremely unreactive fibers on the very surface of the sheet, and this led us into some of these explorations.

Prof. Brecht: With regard to loss of strength on the drying of mechanical pulp, it depends on which of the strength properties you are considering. Our experience has been that if the temperature of drying was not too high and the solids content not too great, the tensile strength could be recovered almost completely by using vigorous agitation on the suspension. In the case of folding strength, however, the decrease brought about by drying was most difficult to recover. Possibly this is of little interest for most purposes. Where high bulk is the criterion, the alterations in properties on drying have little significance. Where high strength is required, some difference is brought about. There is no doubt in my view that the elasticity of the individual fiber is reduced.

High Consistency Screening

A. E. DENTREMONT and R. S. SWENSON

The answer to screening stock at a high consistency has been sought for a long time in the groundwood industry. Investigation of the problem of separating the coarse ground fibers from the accepted fine stock at a high efficiency rate has been deferred because of lack of suitable equipment. Various attempts have been made in the past to use existing equipment to screen at greater than 1% consistency with none of these giving production results good enough to be used in the industry. The reasons for wanting to screen at high consistency are purely economic. The original installations were determined by the tons per day of accepted stock needed in the mill which in turn dictate the number of machines necessary to do this job. A large amount of water is needed in the present-day screens to dilute the stock arriving from the bull screens to a range of 0.5 to 0.7% oven-dry (o.d.) consistency. This thin stock then must be deckered so that it can be stored. Therefore, both the initial installation of screens and deckers must be considered when laying out a full sized mill. If the outlet consistency of the screened stock could be raised threefold, say to 1.5% consistency, the number of deckers could be cut down appreciably along with the number of screens required to do this amount of work. For a mill making upwards of 500 to 600 tons per day of groundwood stock, the equipment on the market today ranges to 60 to 70 tons per day screening capacity and 100 to 125 tons per day on a large decker. Therefore, one third the number of screens, and approximately two thirds the number of deckers would be needed to do this job if screening efficiency could be attained at a high consistency.

With the expansion of the Great Northern Paper Co. at East Millinocket, the use of the selectifier on paper machines proved successful and the possibility of this type of screen for high consistency in the pulp mill was seen. An agreement was made with the Black-Clawson Co. in 1955 to install a 24-in. screen on a trial basis in the groundwood mill. This small screen was operated for a period of nine months under various conditions to see whether this whole idea was feasible. On the basis of these preliminary tests, a larger screen of the same type, being 36-in. in diameter, developed by the Shartle Division, was then installed in a more permanent setup in the screen room (see Fig. 1). Various piping arrangements were made so that either our chemigroundwood stock or the regular groundwood stock could be screened and measured and deckered all in its own system. This production size 36-P selectifier was designed with an initial rating of 100 tons per day capacity. Its installation in our screen room demanded that it be operated on a production basis, therefore, all the pulp screened by it would go to the paper machines. Our operating personnel had to learn its operation and how to handle it under all conditions.

INSTALLATION

The 36-P screen, being of the pressure type, must be kept full at all times. Therefore, a separate pump was provided to keep a positive head on the screen and to minimize the normal pressure variations arising from the bull screen chest. A 75 hp. motor was belted to the drive so that various operating speeds could be studied. The 36-P screen is essentially the same as the 24-P selectifier screen, only larger. The major changes in construction are a convolute spiral inlet and reject spout to aid in putting the stock in circular motion (see Fig. 2). The screen plate is 36 in. in diameter by 42 in. long. The rotor has three impeller blades of the air-foil design to keep the screen plate clean. The cover is of a new locking ring type construction and permits easy removal for inspection, cleaning and impeller clearance adjustment. A suitable air bleed is

A. E. Dentremont, Control Supt., and R. S. Swenson, Pulp Mill Process Engineer, Great Northern Paper Co., East Millinocket, Me.

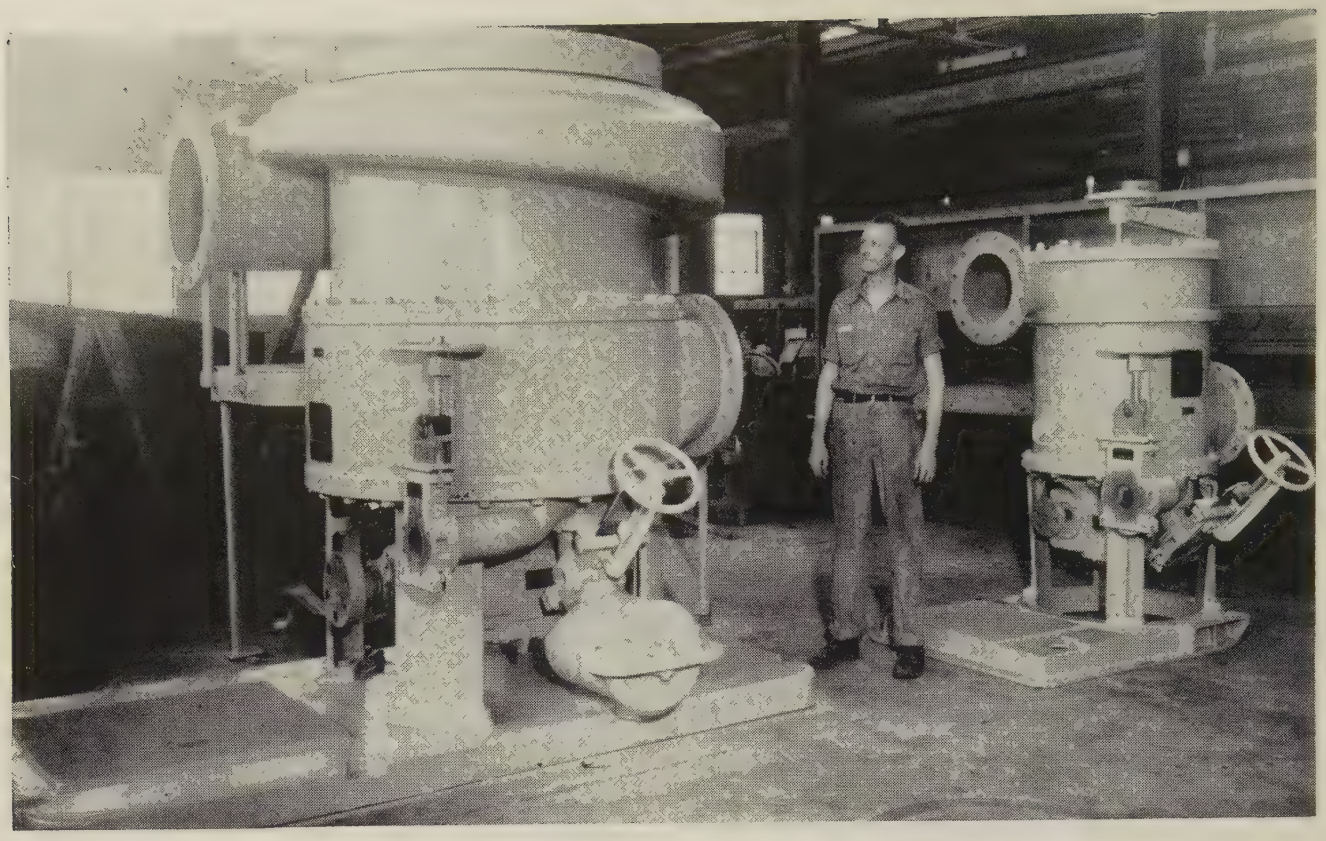

Fig. 1

provided by a $1^1/_2$-in. pipe fitting at the top of the screen. The cover has a slight dome shape to it so that any air build-up in the screen will rise to this portion and can be bled off. It is very important to periodically check the screen for air loading as this will cut the capacity down considerably and then when the stock flow is resumed in this area, a plugging action may occur. Inlet stock to the screen was directly from the bull-screen chest. Jonsson bull-screens with $^3/_{16}$-in. holes are used. From experiments on the smaller screens, it was found that a continuous bleed of rejects is essential for clean accepted pulp. A DeZurik double V-port valve is used to constantly bleed the rejects. When operating at low production rates of 50–60 tons per day, the rejects valve would be throttled down considerably to give the required 10% rejects. With this small open area in the V-port valve, slivers would slowly build up and plug the opening. To alleviate the plugging action, a Black-Clawson timer was installed and an air operator put on the reject valve. This would automatically open the valve wide to purge it, then close down to its original setting on a one-minute cycle. In the big screen we were able to go to a hand-operated, double V-port valve, to control reject flow when the screen operated at 180 tons per day production and 10 lb. pressure in the screen. The open area of the reject valve was approximately $1^1/_4$ sq. in. which was sufficient to pass any of the rejects which would be accumulated in this screen.

When high consistency screening was first begun on the 36-P screen, we experienced excessive plate breakage. Various types of punched plates were supplied by the manufacturer but all failed after comparatively short runs. Apparently great forces are set up in the screen by the action of the bars, so an entirely new approach to the screen by the action of the bars, so an entirely new approach to screen fabrication had to be taken. The result was a centrifugally cast screen plate, $^3/_8$-in. thick. This was drilled to 0.50 or 0.62 in. and then counter-drilled with a 0.090-in. clearance hole from the outside.

CONTROL TESTING

The methods of testing for the control of this screen were made very elaborate since this is a trial installation. A series of pressure taps were installed on the screen with water purging to keep them clear. A manometer was set across the inlet and outlet to give the actual differential pressure across the screen plate. Indicating pressure gages are also on the inlet and outlet sides along with inlet pressure recorder and differential pressure recorder. An orifice plate flowmeter in the accepted line measures the accepted flow and records it on a continuous chart. The amount of rejects are measured by volumetric rise in a tile trough. A separate pump feeds this screen so that its discharge may be throttled, thereby controlling the pressure in the screen. A set of tramp metal traps were installed in the inlet piping to evaluate their effectiveness in removing metal that would otherwise damage the plate. Control of the inlet stock consistency to the screen during production runs was based on frequent sample tests. A white water dilution line in the inlet allowed the operator to main-

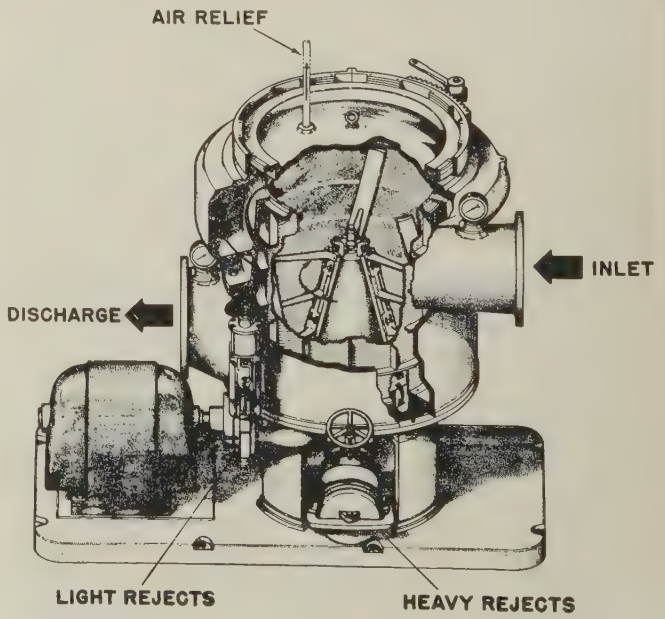

Fig. 2

tain his consistency. Approximately three times a shift the accepted stock was viewed on a blue glass to catch any plate breakage or other reasons for slivers getting by the plate. In our special testing program, a run of samples would be obtained from the inlet, the accepted, and the reject stock. Inspection handsheets, fiber classification, and consistency data were obtained on these stocks. The following data were recorded at the same time: accepted flow, rejects flow, motor amperage, inlet pressure, and differential pressure across the screen plate (see Table I). We found that the most significant tests were the fiber classification retained on the 14-mesh and the handsheets for visual inspection. Since it was run as a production model, however, operation of the paper machines was the final deciding factor. The actual operation of the screen is a very simple matter. Our operators were easily trained to its variables and we had no difficulty in running the screen continuously. This pressure type of screen was found to take normal variations coming from the bull screen chest without adversely affecting the quality of stock in the accepted line.

VARIABLES

In attempting to find the maximum production under certain machine setups, it was obvious that the differential pressure across the screen plate determined the production in tons per day. At inlet consistencies of 1.5%, if the differential pressure rose above 2 or 3 lb. pressure, the accepted stock quality diminished rapidly. We installed an alarm on the differential mercury manometer to sound at 3 lb. differential pressure so that the operator could tell when conditions were arising that would lead to poor quality accepted stock. This worked out very well and in a permanent installation could lead to an automatic control valve on the discharge side, such as a butterfly valve, operated by differential pressure. The size of the screen plate was changed a number of different times to determine the effect on paper quality, and production rates. On the 0.062-in. perforated plate, good running conditions were obtained at 180 tons a day at 1.50% consistency. Differential pressure could be maintained at 2 lb., and a normal swing in consistency taken without any adverse affects in accepted quality. With the smaller screen plate of 0.050-in. perforations, production was found to decrease to approximately 80 tons per day to maintain a low differential pressure, and additional study is needed to get quality accepted fiber. Going from the 0.062 plate to the 0.050-in. plate was a 60% reduction in open area. In studying the effects of consistency, complete runs were made from 0.5 all the way up to 2% consistency on the inlet side. The smaller hole plate resisted the higher consistencies since the hydraulic rate was changed considerably. At 2%, a plugging action was obvious

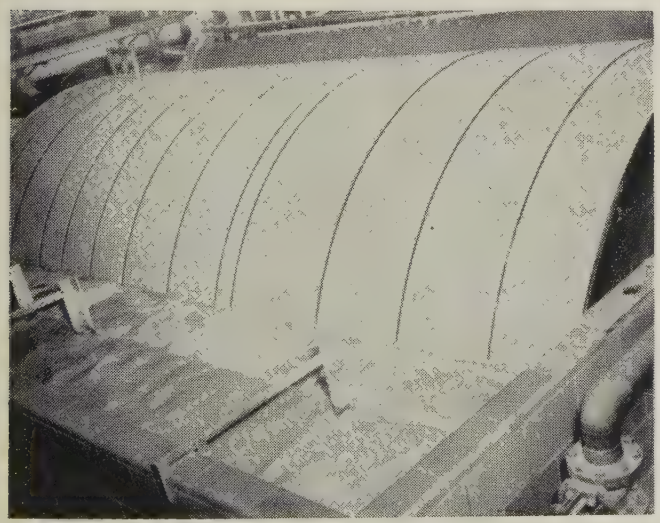

Fig. 3

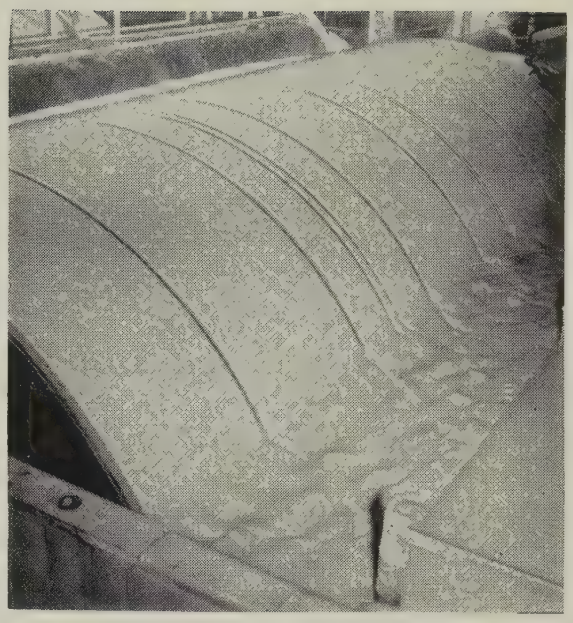

Fig. 4

and the screen could not be operated on continuous duty at this consistency. Variations in consistency during operation would cover a range of 0.80 to 1.70% for periods of 1 hr. with no plugging action. Screening the bull screen stock at the low consistencies of 0.5 to 1% on the inlet basis proved to give poor accepted stock because the rejects were washed through the plate. For this reason we set consistency specifications of 1.3 to 1.6% for the operators to maintain. Various rates of rejects flow were tried and we settled on 10% as being the optimum conditions for this screen running at this consistency. Below 7% rejects on the inlet basis would force slivers through the screen and would not be acceptable pulp. Above 10% too much accepted stock was being passed out the reject pipe. In an effort to reduce the amount of accepted stock in the rejects, a 1-in. fresh water line was added at the discharge of the screen right into the reject spout. This washing effect tended to wash the acceptable stock back into the screen again and proved very successful. The amount of water amounted to approximately 100 g.p.m. and changed the reject consistency slightly. Another variable that was worked from one extreme to the other was the speed of the impeller. This would mean that the swipes per minute per hole of the screen were increased and their effect studied. When higher consistency screening was done, the increase in impeller speed gave better accepted pulp. The clearance between the airfoil bar and the plate was found to be quite critical. With a clearance of

Table I. Operating Data

Screen-plate perforation	0.062 in.
Hub speed	300 r.p.m.
Impeller clearance	0.055 in.
Stock	Groundwood bull-screened stock, 155°F.

	Inlet	*Accepted*	*Rejected*
Flow g.p.m.	2318	2150	168
Consistency (o.d.)	1.53%	1.41%	2.08%
Tons per day	204	183	21
Freeness—Canadian Standard	..	105	238

Rejects on inlet basis	10.3%
Horsepower—instantaneous	59
Inlet pressure	18.5 p.s.i.
Differential pressure across screen plate	2.5 p.s.i.
Deckered stock consistency (o.d.) (couched stock)	8.60%
Deckered stock consistency (o.d.) (sluiced from couch nip)	3.00%
Deckered stock freeness	96

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3×10^{-2} in. at the high spot on the plate, slivers were found to build up on the leading edge between the plate and the bar. This could be detected by the motor load slowly increasing during the operation of the screen. Moving the bars out to 5×10^{-2} in. clearance established optimum running conditions. The further out that the bar is moved the less pulsing action is received from the airfoil and less cleaning is therefore obtained of the perforated hole with each pass of the impeller.

The dewaterizing effect of the plate is noted by the difference in inlet consistency and rejects consistency. The first runs on the screen were made with a 16-gage punched plate. The dewaterizing effect was found to be very high with this thin plate. Feeding $1^1/_2$% on the inlet side of the screen gave upwards of $2^1/_2$ to 3% consistency on the rejects. The dewaterizing effect of the $^3/_8$-in. thick plate was far less than with the 16-gage punched plate. The effects on pulp quality were interesting. The thick plate tended to pass long thin fiber bundles. Perhaps the pipe effect of the long hole had something to do with this observation. It was noted that the amount of short waste in the accepted stock was reduced using the heavier type screen plate.

HIGH CONSISTENCY DECKER OPERATION

The operation of a valveless type decker, $11^1/_2$ by 20 ft., taking 1.5% consistency in the vat is slightly different than operating at low consistencies. Since the water rate of a decker is fixed for best operation, we found it advisable to start up the decker on thin stock and slowly bleed in the heavy stock until a good mat was formed and then thin stock could be cut down and eliminated to maintain the thick mat on the decker. A very heavy mat was obtained on the cylinder when operating at 200 tons per day (see Figs. 3 and 4), and the pressure of the couch roll was cut down from 5 to 1 lb. A large roll of stock accumulated at the nip of the couch and worked its way out to the ends of the couch roll. Special deflector pans were put on the ends to divert this stock (which came off at about 3 to $3^1/_2$% consistency) into the stock chest. Deckered stock off the couch roll came off at approximately 7 to $8^1/_2$% consistency which was the same as that experienced off the low consistency operated deckers. Speed control on a decker is a definite advantage in handling high consistency stock. The effect of less entrained air in the pulp helped appreciably in forming and carrying over a good thick sheet on the decker cylinder and the high groundwood stock temperature of 152°F. also helped in drainage.

CONCLUSIONS

We are still experimenting with high consistency screening and have before us a program of variables to be worked out. However, on the basis of the tests we find that there is a great deal of hope for high consistency screening on a continuous production basis. The chemigroundwood pulp screened very nicely at upwards of 220 tons per day with no operational difficulties encountered. Groundwood screening at 180 tons per day seems to be entirely feasible for newsprint paper. On a decker that is normally rated at 125 tons per day, we have been able to operate at 200 tons per day without any difficulty. Because of its high production, the horsepower requirements per ton are extremely low in this type of operation. We feel that additional work is needed to reduce the amount of accepted fiber in the rejects. This is one of the main drawbacks which we face at the present time. The passage of long thin fiber bundles through the screen plate also presents a problem.

Screening efficiency has not been so good as with our present type of screen and this makes the paper machine screens work harder.

It has been shown, in agreement with current thinking, that the smaller screen plate hole does not necessarily give cleaner pulp. The hydraulic rate of the screen plus inlet consistency as well as per cent rejects all contribute to the end results.

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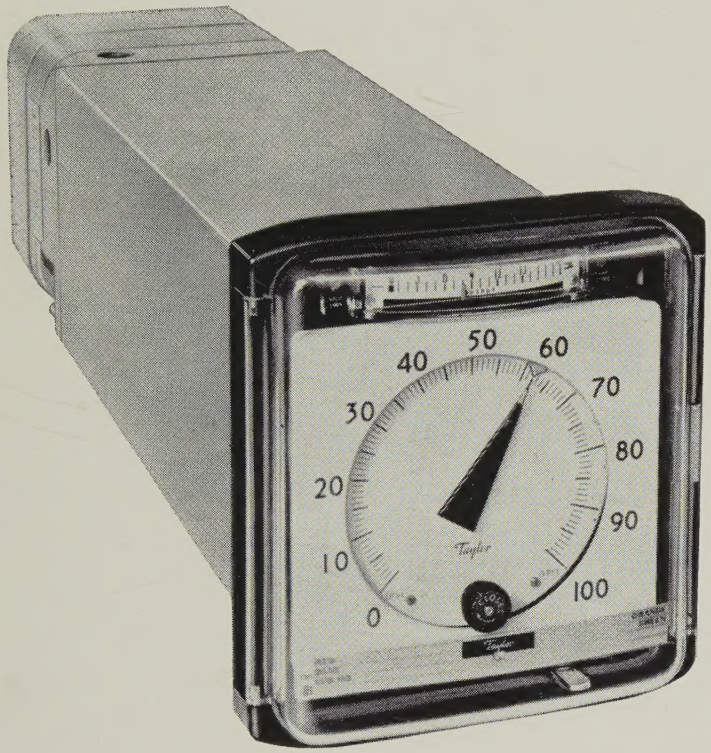

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- Powerful "Servomatic" powered pointers—assure precisely accurate pointer positioning.
- Front-of-panel control response settings give results at a glance.
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- In a control station, continuous indication of set, process and valve pressure.

Optional features include: indication of a second variable on a separate dial; Cascade switch for pneumatic set control; electric or pneumatic process alarms.

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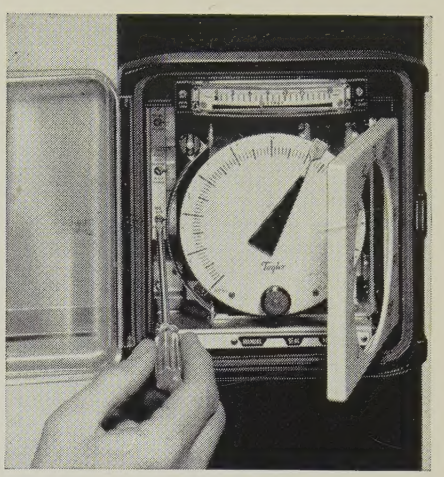
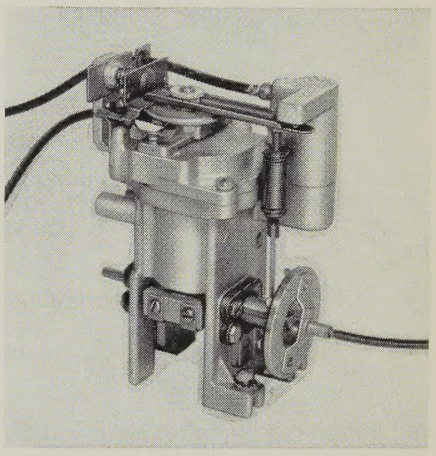


Front of Panel Adjustments. Gain, Reset and Pre-Act* response adjustments can be changed where you see the results while making the change.



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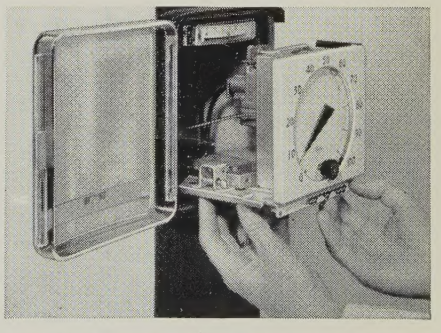

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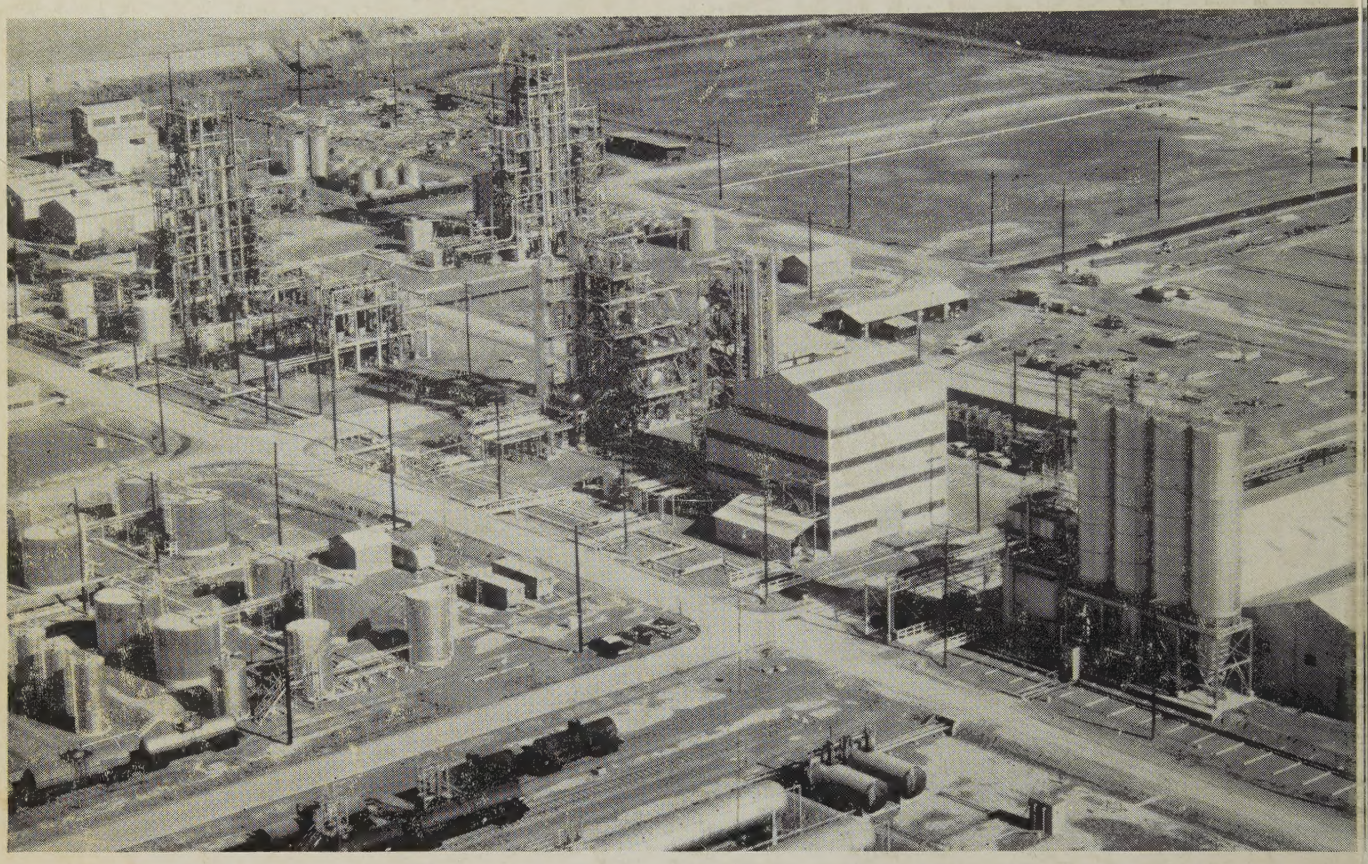

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